



High sensitive and fast formaldehyde gas sensor based on Ag-doped LaFeO₃ nanofibers

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ABSTRACT

Ag-doped LaFeO₃ nanofibers are synthesized by electrospinning method. The chemical and structural characterizations of the composite nanofibers are performed with various techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM) and high resolution transmission electron microscopy (HRTEM). Meanwhile, their formaldehyde (HCHO) sensing properties are investigated in detail. The experimental results indicate that the sensor based on the sample Ag-LaFeO₃ shows excellent gas sensing properties to formaldehyde. The response to 5 ppm formaldehyde is 4.8 at the operating temperature and the sensors have good selectivity. The response and recovery time to formaldehyde gas is 2 s and 4 s, respectively.

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1. Introduction

Formaldehyde (HCHO) is one of the most harmful volatile organic compounds (VOCS) with a high annual production worldwide [1]. Long exposure to the air containing a certain amount of formaldehyde may cause a harmful impact on human health including central nervous system damage, blood, immune system disorders, as well as bronchospasm, pneumonia and respiratory disease [2]. Indoor formaldehyde is often emitted by many products used every day around the house, just like antiseptics, cosmetics, carpet cleaners, glues and adhesives, paper and plastics. Moreover, the wood furniture are the main source of formaldehyde [3]. So it's very important to detect the indoor formaldehyde fast and accurately.

There are several methods reported for formaldehyde detection,

such as chromatography methods [4], catalyzed chemiluminescence methods [5], gas sensor methods [6–8]. Among them, gas sensor is the most suitable method for real-time monitoring [6–8], because gas sensor has the advantage of high selectivity, quick response, stability and low manufacturing cost. The metal-oxides-based gas sensor is one of the most useful instruments to monitor gases. Until now, a great deal of n-type semiconductors have been reported for formaldehyde detection, such as SnO₂ [9], ZnO [10,11], Fe₂O₃ [12], In₂O₃ [13] and MoO₃ [14]. However, research on p-type semiconductors is very scarce, especially on LaFeO₃ [15,16], LaNbO₄ [17], Co₃O₄ [18], V₂O₅ [19], and NiO [20].

Lanthanum ferrite (LaFeO₃) is a perovskite oxide with ABO₃ structure which is a very important material to be used as catalysts [21], electrodes [22], fuel cells [23] and chemical gas sensors [24,25]. LaFeO₃ is a kind of composite metal oxide, and has a good selectivity and high response in the field of gas sensors [26,27]. The synthesis process of LaFeO₃ materials can influence the structure, size and potential applications. Therefore, there are several synthesis methods proposed, including solid state reaction [28], coprecipitation [29], sol-gel [30], hydrothermal method [31] and electrospinning [32].

Nowadays, there are some studies on the LaFeO₃ gas sensors [33–37]. Among these, only a few are used to detect formaldehyde

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[26,33,34]. Besides, the response and recovery time of these formaldehyde gas sensors are a little long, for example, the sensor based on Zn doped LaFeO₃ have the response and recovery time of 100 s and 100 s [26]. In order to improve the response-recovery time, 1 D nanofibers materials are used.

In this work, Ag-doped LaFeO₃ nanofibers were prepared using electrospinning method and characterized by XRD, SEM and TEM. To demonstrate the potential applications, the as-prepared materials were used to fabricate gas sensors. The measuring results revealed that the Ag-doped LaFeO₃ nanofibers exhibited better gas sensing properties than the pure one. According to the experimental results, a possible mechanism involving in the Ag-LaFeO₃ nanofibers gas sensor was also discussed.

2. Experimental

2.1. Synthesis of Ag-doped LaFeO₃ nanofibers

All the chemical reagents used in the experiments were analytical grade and used without further purification. In a typical experiment, 0.207 g lanthanum nitrate (LaFeO₃·6H₂O), 0.193 g ferric nitrate (Fe(NO₃)₃·9H₂O), 4.4 g *N,N*-dimethylformamide (DMF) and 4.4 g ethanol were mixed into a 50 ml beakerflask. After that, a certain amount of Silver nitrate (AgNO₃) (Ag:La = 0%, 2%, 4%, 6% mole ratio) were added into the mixed solution. Then, 0.8 g Polyvinylpyrrolidone (PVP, Mw = 1,300,000) was slowly added into it by magnetic stirring. The precursor solution was subsequently stirred at room temperature for about 5 h to obtain a homogeneous mixture.

The mixture was loaded into a syringe needle for electrospinning and a piece of plat aluminum foil was placed 20 cm away from the needle to collect the fibers. An electrostatic voltage of 18 kV was applied between the needle and the collector. Then, the collected fibers were calcined at 600 °C in air for 2 h, to make sure that all the hydrocarbons in the fibers were fully burned out. After it cooled down to room temperature, the LaFeO₃ nanofibers were collected. The sample LaFeO₃ nanofibers doped by different amounts of Ag were marked as NF0, NF2, NF4, and NF6.

2.2. Characterization of LaFeO₃ nanofibers

The crystalline phase of the prepared samples was analyzed by X-ray diffraction (XRD) which uses a scanning XDS-2000 X-ray diffractometer with Cu K α radiation (λ = 1.5418 Å). Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are used to observe the morphologies and the size of the sample.

2.3. Fabrication and measurement of sensors

The fabricating process of sensors in brief: first, mix the as-synthesized sample and deionized water together with a weight ratio of 100:25 to form a paste. And then spread the paste on a ceramic tube with two Au electrodes and Pt lead wires to form a 300 μm thickness of sensing film. The electrode were fabricated by jetting Au paste on ceramic tube through a metal-jetting system (MJ-10, Beijing Elite Tech Co., Ltd., China). A Ni-Cr alloy coil crossed the tube is employed as a heater to control the operating temperature. The structure of the device is shown in Fig. 1.

A CGS-8 intelligent gas sensing analysis system (Beijing Elite Tech Co., Ltd., China) was used to measure the gas properties. The response value (S) of the sensor was defined as $S = R_a/R_g$, which R_a and R_g represent the sensor's resistance in the absence and presence of the target gases. Meanwhile, the response time (τ_{res}) was the time taken from R_a to $R_a + 90\% \times \Delta R$ ($\Delta R = R_g - R_a$) under the

target gas environment and the recovery time (τ_{rec}) was the time taken from R_g to $R_g - 90\% \times \Delta R$ in the air condition.

3. Results and discussion

3.1. General composition and morphology

The crystal structure of the LaFeO₃ product is characterized by XRD analysis and shown in Fig. 2. All the diffraction peaks pattern can be found to fit to LaFeO₃ crystalline (JCPDS, 37-1493). Except the sample of NF0, there are also some peaks at 38.1°, 64.4° and 77.5° which stand for the (111), (220) and (311) planes of Ag relatively fitting JCPDS, 04-0783 well. Moreover, no additional peaks related to impurities are found, which shows that pure LaFeO₃ or Ag-LaFeO₃ have been synthesized.

SEM images of pure and Ag-doped LaFeO₃ nanofibers with different Ag concentrates are shown in Fig. 3. Although they are different in chemical composition, their morphologies are very similar and they show an image of network from the pictures. Meanwhile, the insert images of Fig. 3 give the picture of single nanofiber and nanocrystalline fibrous morphologies with diameter ranging from 80 to 150 nm are displayed.

In order to obtain more details, transmission electron microscopy (TEM) is employed to analyze the Ag-LaFeO₃ nanofibers. Fig. 4a shows a low-magnification TEM image of single Ag-LaFeO₃ nanofiber and in order to observe the microstructure of the Ag-LaFeO₃ nanofibers, high-magnification TEM (HRTEM) image is shown in Fig. 4b. In the HRTEM image, the interplanar distance of 0.278 nm is close to the d spacings of the (121) planes of the LaFeO₃ nanofibers. In addition, as shown in Fig. 4b, the interplanar distance of 0.236 nm is close to the d spacings of the (111) planes of Ag.

3.2. Gas sensing properties

As it is well known that the sensor's response depends on the operating temperature. In order to find out the optimum working temperature, a series of experiments were performed under a concentration of 20 ppm formaldehyde at different temperature. In Fig. 5, it can be found that the responses of the sensor vary with the different operating temperatures. All the sensors' responses increase and reach the maximum value at 230 °C. The temperature continues to increase, the responses decrease. At 230 °C, the sensors show the best response. So, 230 °C is considered as the optimum operating temperature of the sensors. All the following tests about the sensors' formaldehyde sensing properties are operated at 230 °C.

The response values of sensors to 1, 5, 10, 20, 30, 40, 50, and 100 ppm HCHO at the optimum working temperature are given in Fig. 6. The response increases with the rise of HCHO concentration and tends to become saturated. The sensors' responses to 5 ppm HCHO are 3.56, 4.8, 4.73 and 3.5, which indicate that the minimum detectable concentration limit of HCHO gas is 5 ppm. Compared with pure LaFeO₃ sensor, the Ag-doped LaFeO₃ sensors have better response to HCHO. Among these Ag-doped LaFeO₃ sensors, the response of NF2 sensor is the best to each concentration of HCHO.

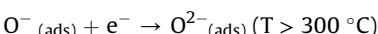
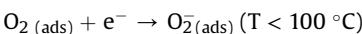
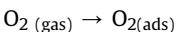
As for gas sensing applications, rapid response and recovery is of great importance. To investigate the response-recovery behaviors of pure LaFeO₃ and Ag-LaFeO₃ sensors, the sensors are exposed to 20, 50 and 100 ppm HCHO at 230 °C, respectively. As is shown in Fig. 7, when the sensors exposed to HCHO, the response increases rapidly and when subjected to air the sensors' recovery to the initial state is also rapid. The response times of NF0, NF2, NF4 and NF6 sensors are all 2 s and the recovery times of these sensors are 5 s, 4 s, 8 s and 6 s, respectively. The rapid response and recovery behaviour can be explained by the 1 D nanofiber structure. The 1 D

structure can facilitate fast mass transfer of the formaldehyde molecules to and from the interaction region as well as improve the rate for charge carriers to transverse the barriers induced by molecular recognition along the fibers [38].

The gas sensing selectivity is also an important parameter to evaluate the sensing ability of sensor. The response of LaFeO_3 and Ag-LaFeO₃ to 100 ppm ammonia (NH_3), toluene (C_7H_8), ethanol ($\text{C}_2\text{H}_5\text{OH}$), formaldehyde (HCHO), acetone (CH_3COCH_3) and xylene (C_7H_8) at their optimal operating temperature are shown in Fig. 8. It can be seen that Ag-LaFeO₃ nanofibers, especially the NF2 ones, have the higher response to HCHO than to other gases compared with LaFeO₃ nanofibers, indicating the better selectivity of Ag-LaFeO₃ nanofibers to HCHO.

3.3. Gas-sensing mechanism

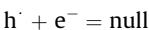
LaFeO_3 is a kind of p-type oxide semiconductor gas sensing materials and its sensing mechanism is based on the changes of resistance in and out of the test gas [39]. When the LaFeO_3 sensors are exposed to air, the oxygen chemically adsorbed on the surface undergoes the following reaction [8]:



The oxygen captures electrons from the materials, leading to the increase in the concentration of holes. Hence, the resistance of the sensors decreases due to the increasing concentration of available carrier. When the sensors are exposed to the reducing gas such as formaldehyde, the reactions take place on the surface of LaFeO_3 oxides as follows [8,40]:



The electrons trapped by the adsorbed oxygen are released. So, a number of electrons are annihilated with holes (h^+) [33]:



Consequently, both the concentration of the holes in LaFeO_3 and conductivity of the sensor decreased. Therefore, the gas response is determined by the changes of the resistance.

Meanwhile, the enhanced sensing properties could be explained by doping with Ag. Noble metal Ag act as a specific adsorption sites to adsorb oxygen and it can activate analyte to facilitate the

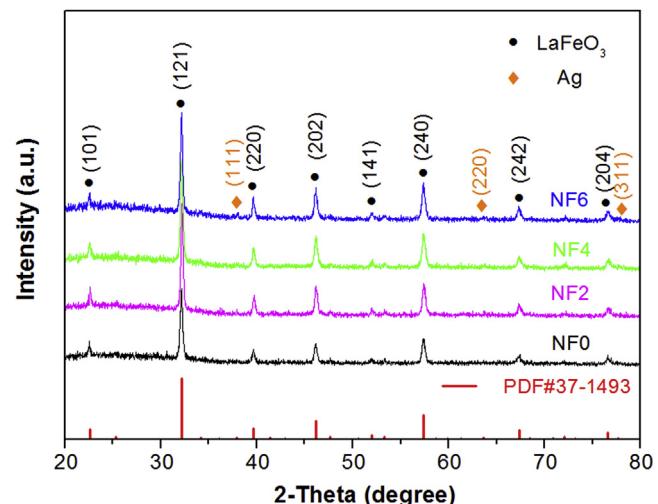


Fig. 2. XRD patterns of LaFeO_3 and Ag-LaFeO₃ nanofibers.

catalytic oxidation on the surface of the sensing materials [41]. Ag nanoparticles facilitate faster adsorption and desorption of the oxygen molecules (O_2) over the sensor surface and capture electrons from support material to become oxygen ions (O^-). This part of Ag can absorb oxygen with the increase of temperature at an appropriate operating temperature and at the same time, it can make the atomic oxygen spread and stick to the surface of the material to form $\text{O}_2(\text{ads})$. Most of Ag acts as a kind of catalyst which can promote the disintegration of formaldehyde into active radicals and increases the reaction between adsorbed surface oxygen ions and formaldehyde gas. Some of them replace the La^{3+} , namely, $\text{Ag}_{\text{La}}^x \rightarrow \text{Ag}_{\text{La}}' + 2\text{h}^+$, which forms the detects [33]. And these detects can provide more holes. As a result, the main charge-carrier concentration increases. And there are also some of Ag filled between the grains of matrix. This part of Ag can enter between the two LaFeO_3 particles and form the Schottky Junction with the LaFeO_3 . So it decreases the contact potential barrier and enhances the interfacial effect. The effect is believed to be good to the sensing performance [42,43]. Thus, the sensing performance is enhanced.

4. Conclusions

In summary, the Ag-LaFeO₃ nanofibers are successfully synthesized via an electrospinning method. Meanwhile, their formaldehyde sensing properties are investigated. The minimum detection limit of the LaFeO_3 sensor can reach 5 ppm at 230°C . The sensors based on Ag-LaFeO₃ nanofibers have better sensing performance and among them, 2% Ag-doped LaFeO_3 has the best

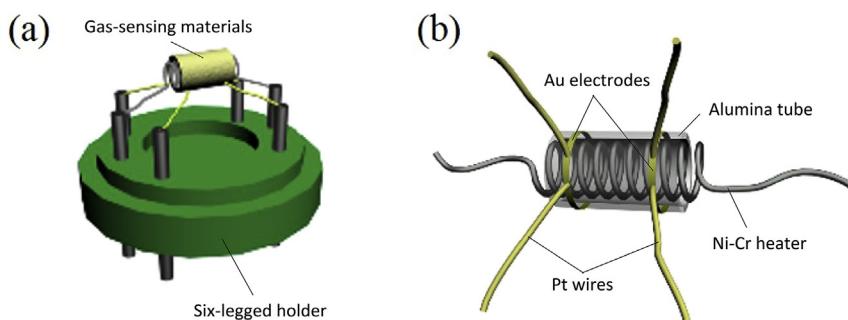


Fig. 1. (a) Device structure of the sensor. (b) The schematic image of the sensor.

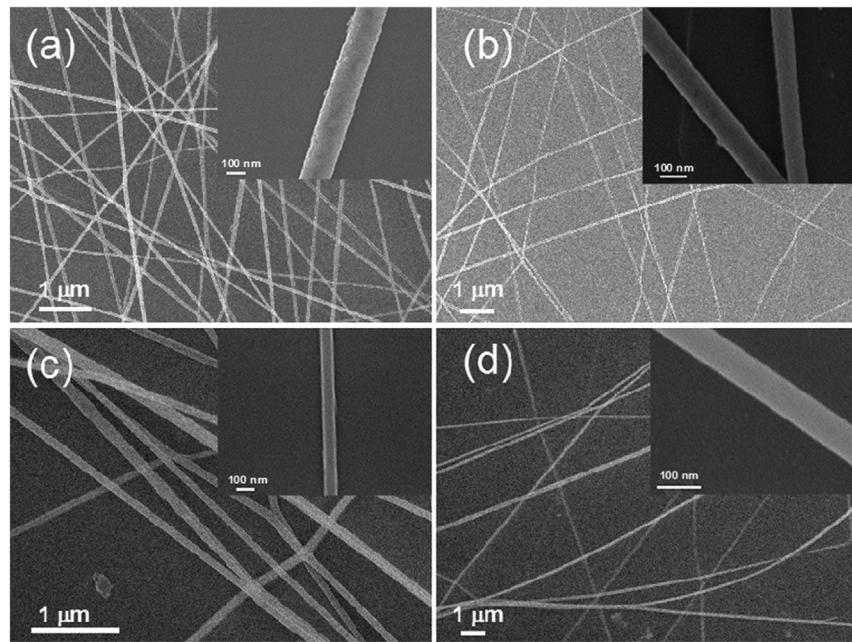


Fig. 3. SEM images of nanofibers. (a)NF0 (b)NF2 (c) NF4 (d) NF6.

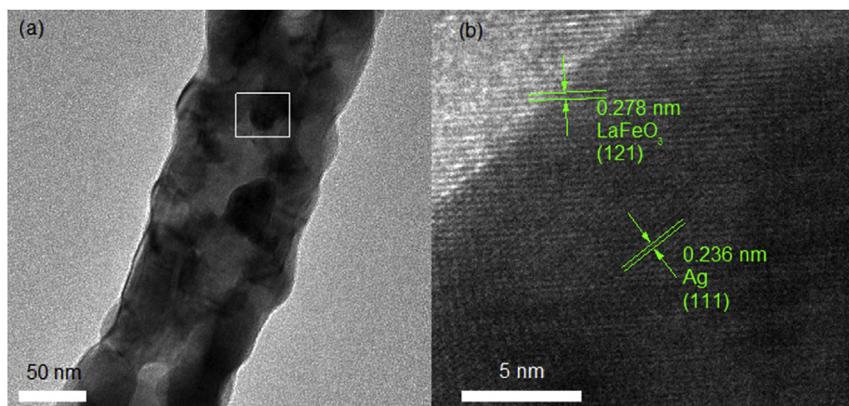


Fig. 4. (a) TEM image of NF2. (b) HR-TEM image of NF2.

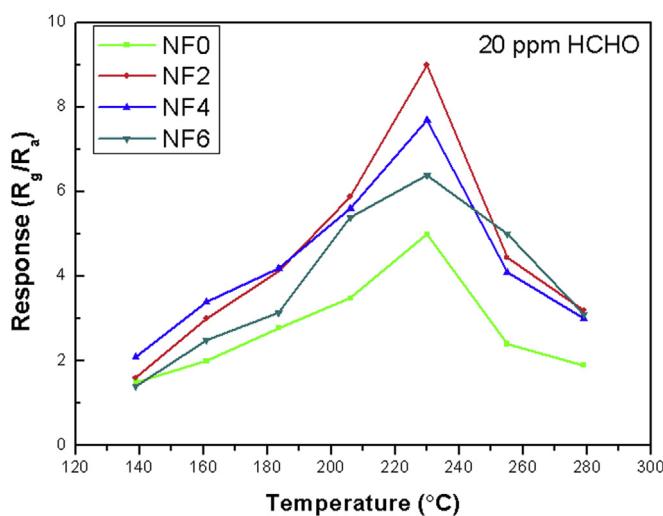


Fig. 5. The sensors' responses to 20 ppm HCHO at different operating temperatures.

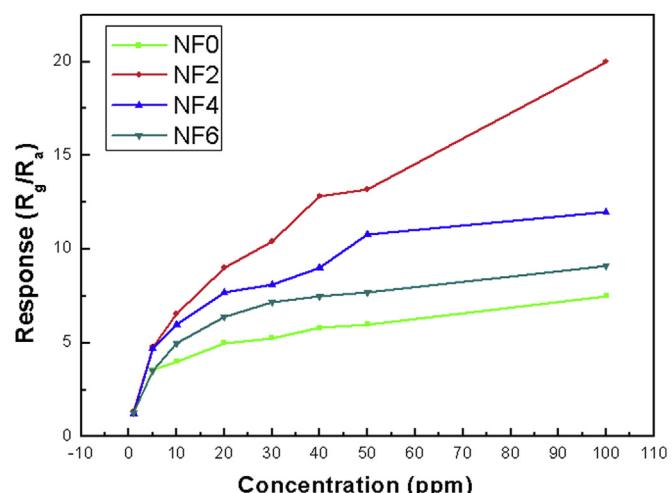


Fig. 6. Curves of sensors' responses versus HCHO concentrations at their optimum operating temperature.

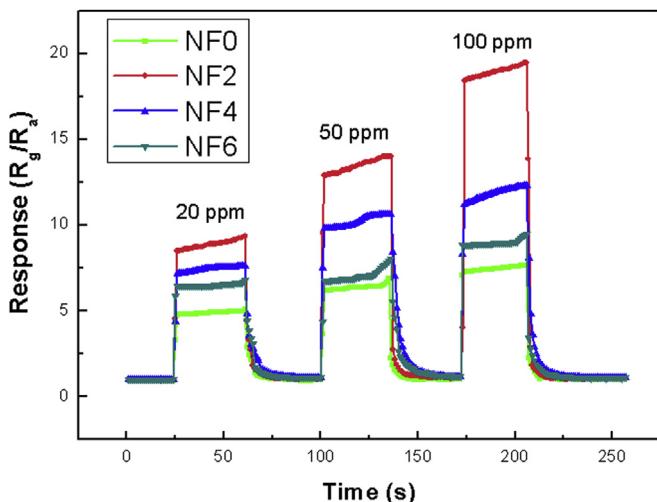


Fig. 7. Transient response of the LaFeO_3 and Ag-LaFeO_3 nanofibers.

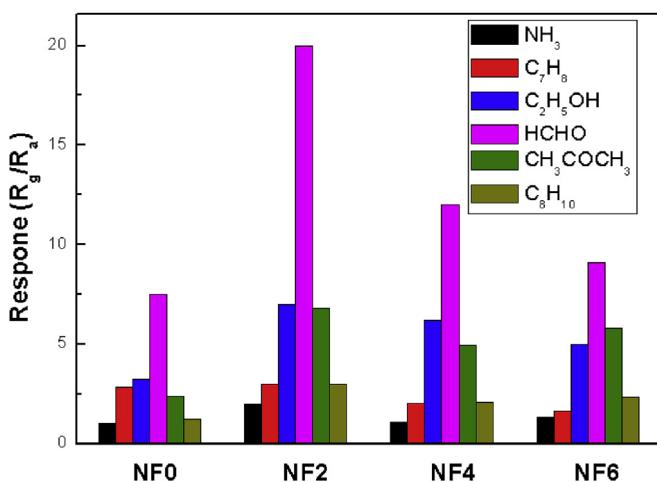


Fig. 8. Responses of the sensors to 100 ppm different gases at their optimum working temperature.

sensing properties. It has the higher response, faster response/recovery speed and better selectivity to formaldehyde compared with the other sensors. The results certify that Ag-LaFeO_3 nanofibers sensor is a potential candidate for high performance formaldehyde sensors.

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