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One-pot synthesis of Au-supported ZnO nanoplates with enhanced gas sensor performance

Jun Zhang [a,*], Xianghong Liu [b], Shihua Wu [b], Bingqiang Cao [a], Shaohua Zheng [a]

[a] School of Materials Science and Engineering, University of Jinan, Jinan 250022, China
[b] Department of Chemistry, Nankai University, Tianjin 300071, China

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ABSTRACT

A novel sensor material of Au nanoparticles-functionalized ZnO nanoplates was fabricated *via* a facile one-pot hydrothermal method. The obtained Au-functionalized ZnO nanoplates were characterized by means of X-ray diffraction (XRD), transmission electron microscope (TEM) and X-ray photoelectron spectroscopy (XPS). Comparative gas sensing studies were carried out on both pristine ZnO and Au/ZnO nanoplates in order to investigate the effect of Au nanoparticles on the sensor performances. Obtained results demonstrated that the Au/ZnO nanoplate sensor exhibited faster response and recovery as well as higher response compared with the pristine ZnO sensor. At 300 °C, the response time of Au/ZnO to 5 ppm ethanol is 13 s, while that of ZnO is up to 135 s. The enhanced sensor performances were attributed to the unique chemical properties of Au nanoparticles and the electronic metal–support interaction. A possible sensing mechanism was proposed for the Au/ZnO nanoplates.

1. Introduction

Chemical gas sensors play a vital role nowadays in many areas such as environmental protection, domestic gas alarms, human health and emissions control [1,2]. Much more research attention is now concentrated on the development of novel sensor materials with advanced performances. Traditionally, gas sensor materials are mainly based on semiconducting metal oxides such as ZnO, SnO_2, WO_3, and Fe_2O_3, although some carbonates materials has also been reported to be sensitive for CO_2 detection [3]. Among the semiconducting oxides, ZnO is one of the earliest [4] and probably the most investigated materials for gas sensor due to its good chemical and thermal stability and high mobility of conduction electrons [5].

It is now well known that the properties or device performances of nanomaterials are highly dependent on their size, shape, morphology and chemical composition. To date, a variety of ZnO nanostructures including nanorods [6,7], nanowires [5], nanofibers [8,9], nanoplates [10], hollow spheres [11] and hierarchical architectures [12,13] have been synthesized and extensively investigated for gas sensing application. The use of nanostructured ZnO could be an effective way toward improving gas sensor performances. For instance, ZnO nanowires have demonstrated great potential for fabricating highly sensitive gas sensors due to the ultrahigh surface-to-volume ratio of one-dimensional nanostructures [5]. In addition, ZnO nanomaterials with a hollow, porous or hierarchical structure have also shown excellent sensor performances in terms of high sensitivity and fast response–recovery, because their loose porous structure could provide a large active surface area and facilitate the gas diffusion and transport in the sensing layers [11–15]. Besides the control over the morphologies of sensor materials, surface modification by noble metals can also provide an alternative method to improve sensor properties [16]. For example, through surface functionalization by noble metals such as Au, Pt and Pd as dopants or promoters, the sensor performances of one-dimensional ZnO [17,18], SnO_2 [19], WO_3 [20,21] and CeO_2 [22] nanostructures, Fe_2O_3 particles [23] and SnO_2 hollow spheres [24] can be significantly enhanced. Especially, the metal–ZnO hybrid nanomaterials such as Au/ZnO nanopyramids [25], nanorods [18,26,27] and core–shell nanoparticles [28], Ag/ZnO microspheres [29] and nanocrystals [30], Pd/ZnO nanowires [17] and Pt/ZnO nanoflowers [31] and nanowires [32] have demonstrated great potential for applications in gas sensing, optics, solar cells and photocatalysis. For fabricating these noble metal-functionalized sensor materials, at least two steps are involved, including the first-step synthesis of support materials and then subsequent functionalization of noble metals using a second step, which makes the synthesis process quite complex and not economic. Hence it is very necessary to establish an efficient and facile route to fabricate noble metal-functionalized sensing materials.

In this work, we develop a simple one-pot hydrothermal method for the preparation of Au-functionalized ZnO nanoplates, and their

* Corresponding author.
E-mail address: zj1025@gmail.com (J. Zhang).

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gas sensor performance have been evaluated by using ethanol as the main probe molecules. It is found that Au functionalization lead to prominent promoting effect on the sensor performances. The Au-supported ZnO nanoplates sensor exhibits significantly enhanced device performances in terms of fast response speed and high response in comparison to pristine ZnO. The possible sensing mechanism was discussed for the enhanced Au/ZnO sensor.

2. Experimental

2.1. Chemicals

Chemicals such as Zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$), NaOH and absolute ethanol were of analytical grade and purchased from Guangfu Fine Chemical Research Institute (Tianjin, China). $HAuCl_4{\cdot}4H_2O$ was obtained from Yingdaxigui Chemical Reagent Company (Tianjin, China). Distilled water was used throughout the experiments.

2.2. Synthesis of Au/ZnO nanoplates

Typically, 0.01 mol of $Zn(CH_3COO)_2{\cdot}2H_2O$ and 0.1 mol of NaOH was dissolved into a mixture containing 35 mL of H_2O and 40 mL of ethanol under stirring. Subsequently, 5 mL of $HAuCl_4$ (0.01 M) aqueous solution was added into the mixture. After further stirring for 1 h, the stock solution was transferred into a 100 mL Teflon-lined autoclave and maintained at 120 °C for 12 h. After reaction, the purple precipitate was collected by centrifugation and washed several times with distilled water and ethanol, and dried at 80 °C. To examine the effect of Au on the sensor properties, pristine ZnO was also prepared without the addition of $HAuCl_4$ and tested for gas sensing.

2.3. Characterizations and gas sensing test

The products were characterized by means of powder X-ray diffraction (XRD, Rigaku D/max-2500, Cu Kα, λ = 1.5418 Å), transmission electron microscope (TEM, Philips FEI Tecnai 20ST, 200 kV), X-ray photoelectron spectroscopy (XPS, Kratos Axis Ultra DLD spectrometer, Al Kα X-ray monochromator). Gas sensing tests were performed on a commercial HW-30A Gas Sensing Measurement System (HanWei Electronics Co., Ltd., Henan, China) at 300 °C at a relative humidity of 22–37% (under ambient condition, ca. 15 °C). Test gas such as ethanol was injected into the testing chamber on HW-30A by a microsyringe. The sensor response is defined as the ratio of R_a/R_g, where R_a and R_g are the electrical resistance of the sensor in air and in test gas, respectively. The response time is defined as the time for the sensor to reach 90% of response amplitude. Details of the sensor fabrication, photograph and gas sensing test principle can be seen in our previous publications [12,13,24].

3. Results and discussion

3.1. Characterization

The Au-supported ZnO nanoplates have been fabricated *via* a simple one-pot hydrothermal procedure, where the formation of Au nanoparticles and crystallization of ZnO support are occurred simultaneously. Fig. 1 shows the schematic one-pot procedure for the synthesis of Au/ZnO nanoplates. In the reaction system, the concentration of OH^- is largely excessive compared to Zn^{2+}. $Zn(OH)_4{}^{2-}$ species undergo dehydration and give rise to ZnO. Due to the reductive property of ethanol, Au nanoparticles might be formed by the reduction of $AuCl_4{}^-$ by ethanol.

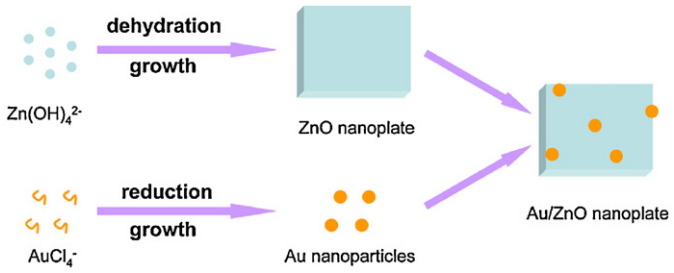

Fig. 1. Schematic procedure for the synthesis of Au/ZnO nanoplates.

The crystalline phase of the samples was determined by XRD. Fig. 2 shows the XRD patterns of pristine ZnO and Au/ZnO materials. All the diffraction peaks of ZnO can be indexed to hexagonal wurtzite ZnO (JCPDS 36-1451) and no peaks for any other phase or impurities could be detected. By comparison, it is observed that the XRD pattern of Au/ZnO is very similar to that of ZnO, indicating that the formation of Au in the hydrothermal reaction process has no influence on the crystal structure of ZnO. Besides the diffraction peaks of ZnO, the XRD pattern of Au/ZnO also presents three small peaks at 38.2°, 44.5° and 64.6°, which can be ascribed to the (1 1 1), (2 0 0) and (2 2 0) planes of face-centered cubic (fcc) Au (JCPDS 01-1174), respectively.

The morphology and microstructure of the Au-supported ZnO nanoplates were observed by TEM and the images are shown in Fig. 3. From Fig. 3a, it is seen that the ZnO particle possesses a plate-like morphology. The ZnO nanoplates have a diameter of 150–350 nm and thickness of ca. 56 nm. Furthermore, Au nanoparticles can be seen supported on the surface and edge of ZnO nanoplates, as indicated by the white arrows. Fig. 3b displays a magnified TEM image. In this image, Au nanoparticles with a size of 15–36 nm can be clearly seen on the surface of ZnO nanoplate.

XPS is a very useful technique to analyze the surface composition of materials and has been employed to check the chemical composition of the Au/ZnO nanoplates. From the wide XPS spectra shown in Fig. 4a, it is confirmed that the sample is of high purity, as only Au and Zn elements have been detected. Fig. 4b–d displays the high-resolution XPS spectra of Zn 2p, Au 4f and O 1s, respectively. The binding energy at 1021.2 and 1044.3 eV in Fig. 3b are assigned to Zn $2p_{3/2}$ and Zn $2p_{1/2}$ of Zn^{2+}. The XPS spectrum of Au 4f in Fig. 4c exhibits two signal peaks at 82.9 and 88.1 eV corresponding to Au $4f_{7/2}$ and Au $4f_{5/2}$. In Fig. 4c, two interference peaks due to Zn 3p are also seen to overlap with Au $4f_{5/2}$, which cause a positive shift in the binding energy of Au $4f_{5/2}$ from 87.4 eV of bulk Au to 88.1 eV.

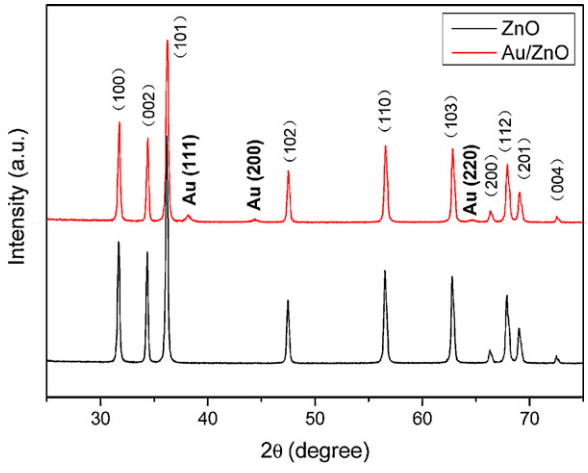

Fig. 2. XRD patterns of ZnO and Au/ZnO nanoplates.

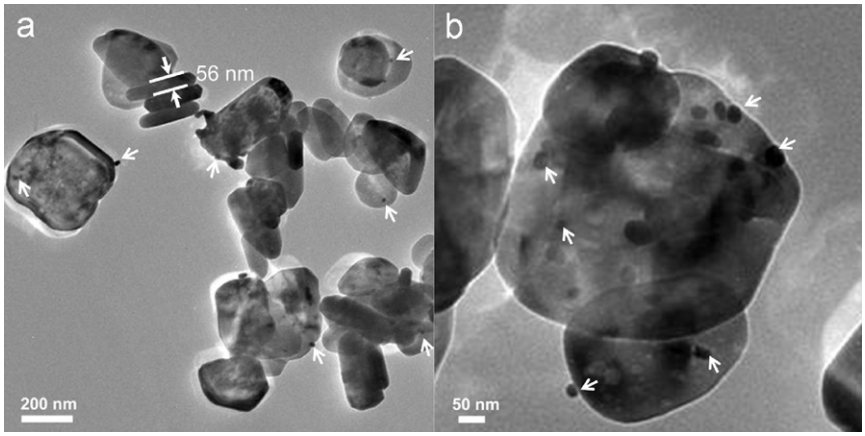

Fig. 3. TEM images of the obtained Au/ZnO nanoplates.

However, it is noted that binding energy of Au $4f_{7/2}$ shows a negative shift of 0.9 eV in comparison to 83.8 eV of bulk Au, which is probably caused by electron transfer from the conduction band of ZnO to Au due to the metal–support electronic interaction [29,33,34]. According to previous publications, the negative shift in the binding energy of Au $4f_{7/2}$ is probably induced by two reasons: the small size effect of Au nanoparticles (<1 nm) and the metal–support interaction [33–35]. However, the size of the Au nanoparticle supported on ZnO nanoplates (Fig. 3) is obviously much larger than 1 nm. So the negative shift in the binding energy of Au 4f should be mainly ascribed to the metal–support interaction. It is important to mention that the so-called metal–support interaction has been thought as a promotion factor in improving the catalyst performances in both low temperature CO oxidation [34,36,37] and photocatalysis [38]. The XPS spectrum for O 1s in Fig. 4d has been fitted by Gaussian method to three peaks at 529.9, 531.5 and 533.2 eV, which can be ascribed to O^{2-} of lattice oxygen, OH^- or O^- of surface-adsorbed oxygen and $O_2{}^{2-}$ of surface-chemisorbed oxygen [39,40]. Furthermore, the mass content of Au in the nanocomposite is determined to be 1.1% by XPS analysis.

3.2. Gas sensing performance

Although various metal–ZnO hybrid nanostructures have been investigated for device applications [17,18,30], there has been rare report on the device properties of Au/ZnO nanoplates. Therefore it

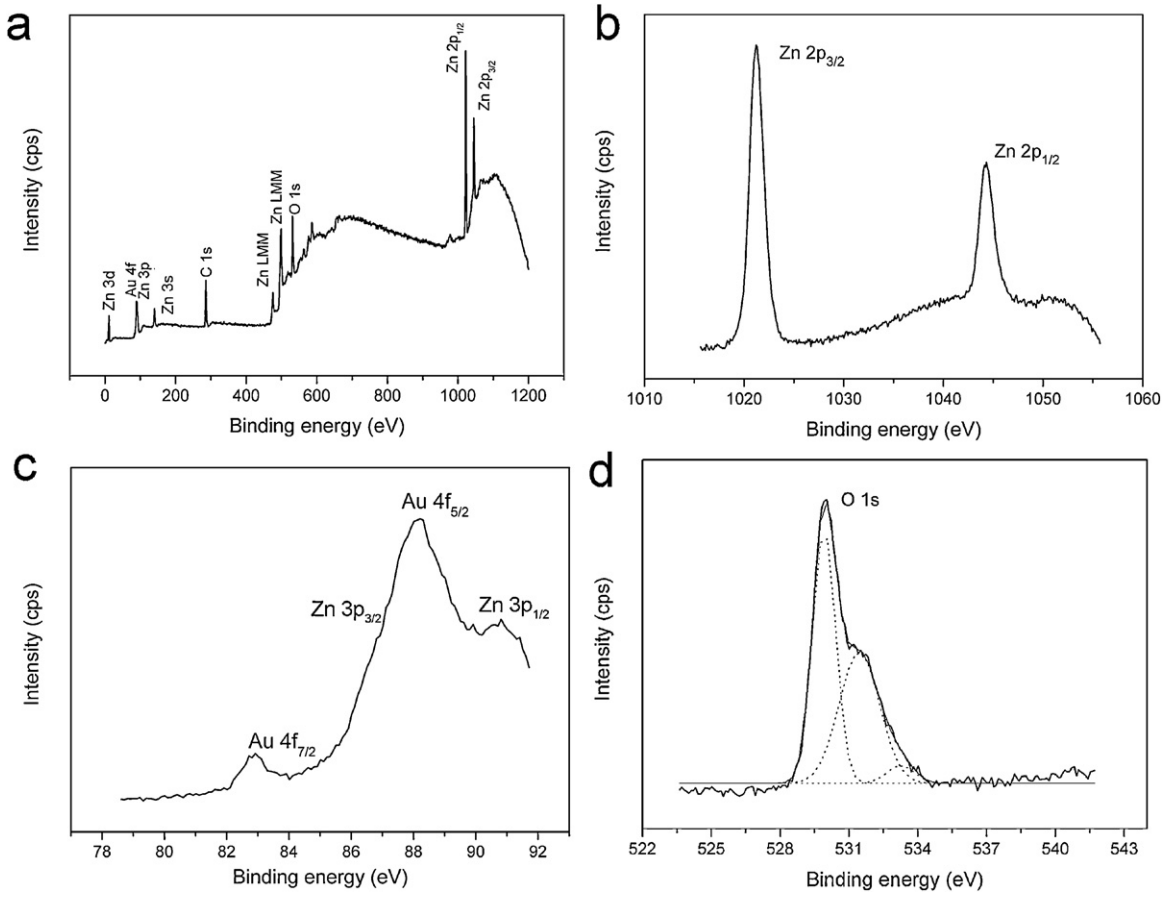

Fig. 4. (a) Wide XPS spectrum and high resolution XPS spectra of (b) Zn 2p, (c) Au 4f and (d) O 1s of Au/ZnO nanoplates.

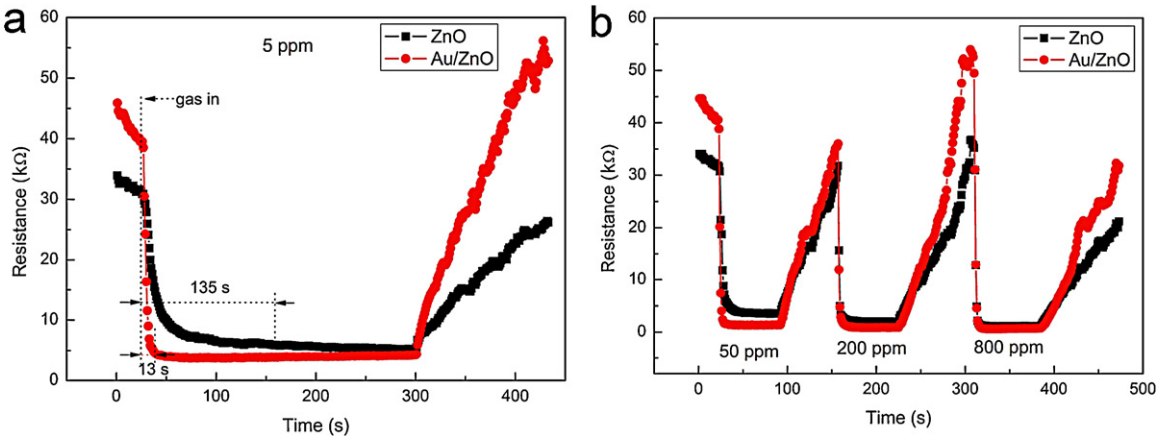

Fig. 5. Dynamic response–recovery curves of ZnO and Au/ZnO nanoplates to (a) 5 ppm and (b) 50, 200 and 800 ppm ethanol.

is of great interest to investigate the obtained Au-supported ZnO nanoplates for gas sensing.

Fig. 5a displays the dynamic response–recovery curves of ZnO and Au/ZnO for sensing 5 ppm ethanol. It is observed that the two curves show significant difference in their response and recovery speed, which is probably caused by the functionalization of Au nanoparticles. When the sensors are exposed to ethanol gas, the resistance of Au/ZnO sensor shows a dramatic and sharp decrease, while the pristine ZnO sensor shows a quite slow response. The reduction of sensor resistance on exposure to reducing gases is characteristic of n-type semiconductor metal oxide sensing materials. In Fig. 5a, the response time of Au/ZnO sensor is determined to be 13 s, much shorter than that (135 s) of the ZnO sensor. It is also seen that the Au/ZnO sensor recovers faster than pristine ZnO does. The effect of gas concentration on the sensor responses was also investigated. Fig. 5b compares the sensing performances of the two sensors for detecting 50, 200 and 800 ppm ethanol. It can be seen that the Au/ZnO sensor has a higher response than ZnO to each concentration. The Au/ZnO sensor also possesses a faster response speed compared with pristine ZnO, though their recovery speeds show little difference. Fig. 6 exhibits the responses of the two sensors to different ethanol concentrations. It is observed that the Au/ZnO sensor possesses much higher response values in comparison to pure ZnO.

Since the test conditions are identical for the two sensors, the differences in the sensing curves presented by the two sensors hence should be directly related to the supported Au nanoparticles. It has been widely accepted that the use of noble metals as dopant or sensitizer can improve sensor performance. Noble metal dopants could modify the sensing reactions between reducing gases and absorbed oxygen species (O_2^-, O^- and O^{2-}) [41–43], mainly due to the spillover effect of catalytic noble metals or the formation of Schottky barrier at the interface between metal and semiconductors [1,16,44]. Au nanoparticles may catalyze and accelerate the kinetics of sensing reactions, leading to a faster response speed of the sensor. ZnO is known to be a surface surface-depletion controlled type of sensor materials. The surface absorbed oxygen species (O_2^-, O^- and O^{2-}) could produce a depletion layer on the surface of ZnO by extracting electrons from the conduction band of ZnO (Fig. 7a and b), which reduces the concentration of charge carriers in the sensing layers and leads to an increased resistance or decreased conductivity of the sensor [42]. Furthermore, the metal–support interaction which is also proved by the XPS result might result in the electron transfer from ZnO to Au (Fig. 7c) [17,29]. In this regard, the noble metal nanoparticles may serve as electron sinks [29]. This kind of interaction could further widen the electron depletion layers on the surface of ZnO (Fig. 7d), hence further changes the resistance or conductivity of the sensor [17,18,21].

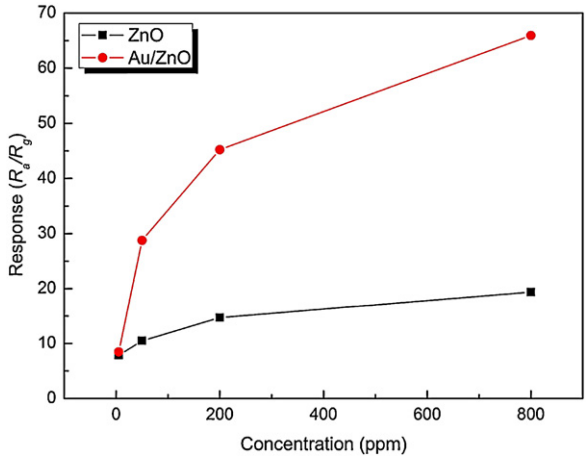

Fig. 6. Sensor responses of ZnO and Au/ZnO nanoplates to various ethanol concentrations.

To further examine the sensing ability of the obtained Au/ZnO nanoplates, the reproducibility of the sensor was also tested. Fig. 8 shows the dynamic sensing curves of Au/ZnO exposed to four cycles of successive tests for 100 ppm ethanol. From the picture, it can be seen that sensor shows very good stability, as the four-cycle sensing curve only shows neglectable change. In addition, several other

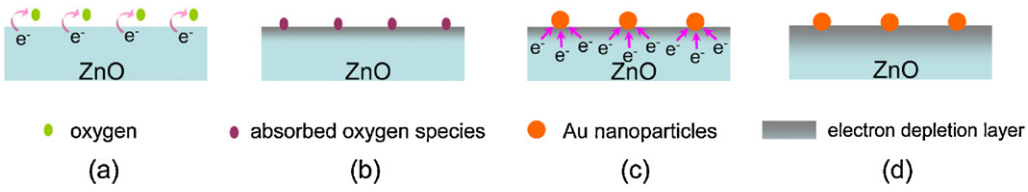

Fig. 7. Proposed sensing mechanism for the Au/ZnO sensor.

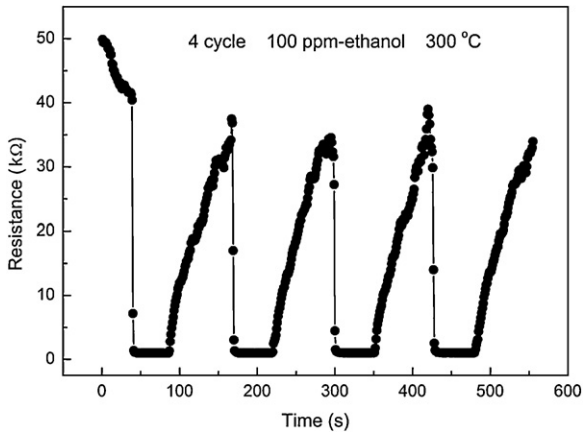

Fig. 8. Reproducibility of Au/ZnO nanoplates for successive detection of 100 ppm ethanol.

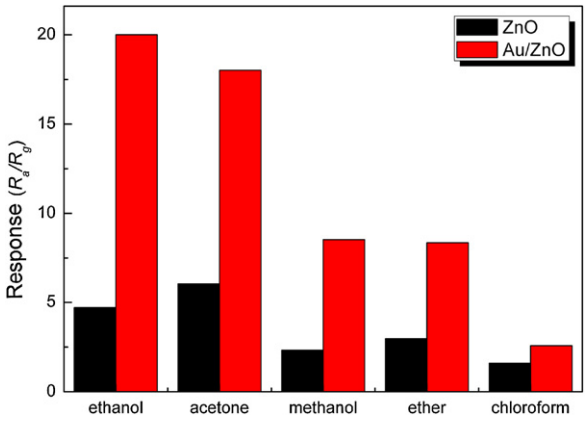

Fig. 9. Sensor responses of ZnO and Au/ZnO nanoplates to various gases (100 ppm).

gases have been tested to investigate the selectivity of the Au/ZnO sensor, and the results are shown in Fig. 9. It is noted that without functionalization of Au nanoparticles, the pristine ZnO sensor has the highest response to acetone, while the Au/ZnO sensor exhibits the highest response to ethanol, indicating the Au/ZnO nanoplates are more sensitive to ethanol and have a potential for developing ethanol sensor.

4. Conclusion

In summary, a novel functional sensor material of Au-supported ZnO nanoplates has been successfully synthesized *via* a simple one-pot hydrothermal procedure. Obtained results demonstrate the surface functionalization of Au nanoparticles could significantly improve the sensor performance with respect to faster response and recovery speed, higher sensor responses and selectivity. The enhanced sensor properties are ascribed to the synergic Au/ZnO interaction and unique chemical properties of Au nanoparticles. The easy synthesis procedure reported in this work is expected to provide potential for fabricating other noble metal-based functional nanomaterials.

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Biographies

Jun Zhang obtained his PhD degree from the Department of Chemistry, Nankai University in 2011. His research is focused on the synthesis and applications of functional nanomaterials.

Xianghong Liu is now studying for her PhD at Nankai University with a research interest covering gas sensor and catalyst materials.

Shihua Wu is professor of Department of chemistry, Nankai University, where he leads a research group working on the synthesis and characterization of nanomaterials for chemical gas sensors and catalysts.

Bingqiang Cao is a Taishan Scholar Endowed Professor of School of Materials Science and Engineering, University of Jinan. His research group focuses on the development of micro-nanomaterials for application in optical-electronics, gas sensor and so forth.

Shaohua Zheng is professor of School of Materials Science and Engineering, University of Jinan. His research is related to synthesis of micro/nano-powder materials for antifriction applications.