



Gas sensing properties of SnO₂ nanoparticles mixed with gold nanoparticles

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Abstract: SnO₂ nanoparticles mixed with different amounts of gold nanoparticles (GNPs) were synthesized and their CO sensing properties were investigated. The sol–gel method was employed to prepare the initial solution. SEM, TEM, XRD, DLS and spectrophotometry were used to characterize the nanoparticles. The pure sensors showed a response of about 4 to 12.8 for (20–80)×10^{−6} CO at operating temperature of 340 °C. The response and recovery time at 50×10^{−6} Co is about 10 and 14 s, respectively. The amount of GNPs optimized was used to create high performance GNP–SnO₂ sensors ($m(\text{Au})/m(\text{Sn})=3.7663\times10^{-4}$) and optimal operating temperature was about 260 °C and the response at concentrations of (20–80)×10^{−6} was 8.3 to 29.5, respectively.

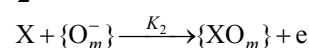
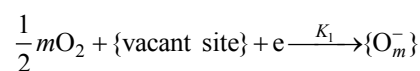
Key words: GNP–SnO₂; SnO₂; nanoparticles; gas sensor; sol–gel

1 Introduction

Gas sensors based on metal oxide semiconductors are widely used to detect combustible, toxic, and pollutant gases which can be harmful to the environment. A change in gas combination around a semiconductor affects its electrical conductivity. Different metal oxide semiconductors have been suggested for gas detection. Metal oxide nanostructures such as tin oxide, zinc oxide, titanium oxide, indium oxide, and tungsten oxide [1–9] along with additive materials such as Ag [10], Pd [1], Cu, and In, are the most common resistive gas sensors which are used to reveal gases such as CO at various temperatures. In recent years, tin oxide gas sensors have been highly regarded for their use in environmental pollution monitoring [11–13]. SnO₂ in the form of bulk or thin film as a wide band gap n-type semiconductor ($E_g=3.6\text{--}3.8\text{ eV}$) is widely used in gas sensors, optical electronics, and catalysts because of its special chemical and physical properties. These characteristics of SnO₂ depend on its structural parameters. For instance, the gas sensing properties of SnO₂ strongly depend on particle size and surface area. Decreasing the size of the nanoparticles increases the surface area and sensor efficiency [14].

Semiconductor gas sensors work based on electrical change of semiconductor surface. Sensor specifications

vary at different temperatures. Because the sensitivity of semiconductor sensors at room temperature decreases, researchers have increased the operating temperature of the sensors to allow them to work properly [1,15–19]. Gas sensors produced by thin or thick film technology operate at 100 to 500 °C [1,14,20–22]. SnO₂ film at high temperatures due to the high electron affinity of oxygen atoms eliminate or trap electrons when there is a lack of oxygen atoms or in the presence of extra oxygen atoms. The absorption of reducing gas molecules like CO, through reacting with oxygen ion species to form CO₂, tends to release trapped electron and SnO₂ nanoparticle conductivity [12]. The working mechanism of the gas sensors based on semiconductor metal oxides such as SnO₂ is



Some methods of preparing SnO₂ nanoparticles include the sol–gel [2,20–22] and hydrothermal methods [3]. The sol–gel process is a chemical method of producing metal oxide nanoparticles. Sols are particles of 1 to 100 nm in diameter which are immersed into the solution. Because they show slight Brownian movement, they remain suspended in the solution. Gel is a solid

adherent network with sub-micrometer pores and polymeric chains. In the present study we produced SnO₂ nanoparticles having high dispersion and low diameter size using the sol–gel method [23,34]. Besides, GNPs synthesized and mixed with SnO₂ Nanoparticles. Then, we optimized the amount of mixing agent for the GNP-SnO₂ sensors. The best operating temperature for sensors was achieved and the response, response time of the GNP-SnO₂ and pure SnO₂ sensors were analyzed under different conditions.

2 Experimental

The current study fabricated and investigated pure SnO₂ and GNP-SnO₂ gas sensors. Different experiments were then carried out to compare their performances.

2.1 SnO₂-nano particle preparation

For preparation of SnO₂ sol, 6.2 mL SnCl₄ and 23.865 mL 1-propan (C₃H₇-OH) were poured into a closed container and mixed rigorously. In another container, 2.865 mL H₂O and 11.425 mL 1-propan were mixed. The two solutions were then combined and mixed thoroughly. Next, 5.715 mL H₂O and 24.435 mL 2-propan (2-C₃H₇-OH) were mixed and added to the existing solution and the resulting solution was blended for 2 h.

2.2 Preparation of GNP-SnO₂

For preparation of GNP-SnO₂, the GNP solution was prepared by sodium citrate reduction of gold chloride in an aqueous solution. The GNPs were encapsulated in 4-methylbenzenthioi synthesized as follows. Initially, 10 mL of 0.0288 mol HAuCl₄·3H₂O aqueous solution was poured into a container. Next, 20.6 mL of 0.0358 mol toluene [N(C₈H₁₇)₄Br] was added to the container and stirred for 15 min. Then, 23.8 mL of 0.0139 mol 4-methylbenzenthioi (HS-C₆H₄-CH₃) and 8.25 mL of 0.3836 mol NaBH₄ in the form of an aqueous solution were stirred into the solution in the container and mixed for 3 h.

The solution formed separate black/brown phases. To separate the GNPs from the solution, ethanol was added to increase the volume of the solution, which was then frozen for 12 h. The black sediment was filtered using a 0.2 μm PTFE filter [35]. The sizes of the GNPs were 2.4 to 4.4 nm with Gaussian size distribution. Different amounts of GNP solution were added to the SnO₂ sol and were mixed by magnetic stirring for about 3 h to prepare a solution containing varying amounts of GNPs.

Samples 1–5 were produced by adding 0.0125, 0.0083, 0.00625, 0.0041 and 0.00312 mL GNP solution to 1 mL pure SnO₂ sol, respectively. The mass ratio of Au/Sn was 1.1482×10^{-3} for sample 1 to 2.8477×10^{-4} for

sample 5.

2.3 Test set-up

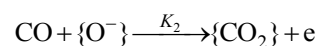
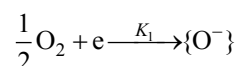
Alumina ceramics with 1 mm thickness were sliced into 1.6 cm × 1 cm pieces and platinum interdigitated contacts having 1 mm gaps and 10 fingers with 1 cm in length were DC sputtered onto the ceramic substrate. The ceramics pieces were dipped into the solution for 30 min. After dipping, the prepared sample was sintered at 600 °C for 30 min.

The experimental set-up consisted of a glass tube reactor, a heater with 400 W electrical power, and a data acquisition system for continuous monitoring of sensor response. The set-up had gas inlet, gas outlet and mass flow controller. The temperature of the sensors was varied from 100 to 400 °C.

XRD analysis was performed using a Philips X'Pert MPD instrument with Cu K_α radiation ($\lambda=1.54178 \text{ \AA}$) at 40 kV/30 mA. Hitachi S4160 model field emission scanning electron microscope was used for FESEM imaging. The response was defined as the ratio of sensor resistance in dry air to sensor resistance in the detected gas ($R_{\text{air}}/R_{\text{gas}}$). The response time was the length of time required for sensor resistance to arrive at 90% modification. Recovery time was defined as the time required for 60% variation to gain the initial content in the air.

3 Results and discussion

The GNPs used as catalyst with specific size and shape have additional photo catalysis effect under visible light [36]. The GNPs supported on the SnO₂ nanoparticles exhibited higher catalytic activity when illuminated with visible light. So, CO molecules could be oxidized more easily by lowering the activation energy of the following reactions which tend to release the trapped electrons to the body and increase conductivity.



Visible light can enhance local electromagnetic fields, heat, and activate GNPs by local surface plasmon resonance (LSPR) effect. This lowers the activation energy of the reactions. Figure 1(a) shows the absorption spectra of the GNP solution. As shown, GNPs have high absorption in UV spectrum and have a local maximum at 550 nm (visible light).

The size distribution for the GNPs was obtained using DLS technique as shown in Fig. 1(b). The size of the GNPs was 2 to 3 nm. The shape of the nanoparticles was investigated by TEM and XRD as shown in Figs. 2(a) and (b), respectively. A TEM core size of

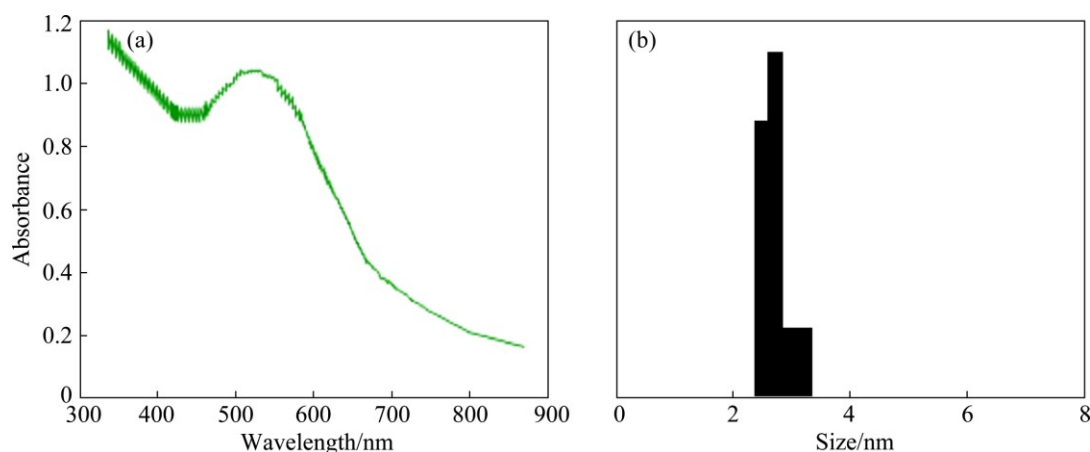


Fig. 1 Absorption spectra of GNP solution (a) and GNP size distribution (b) through DLS

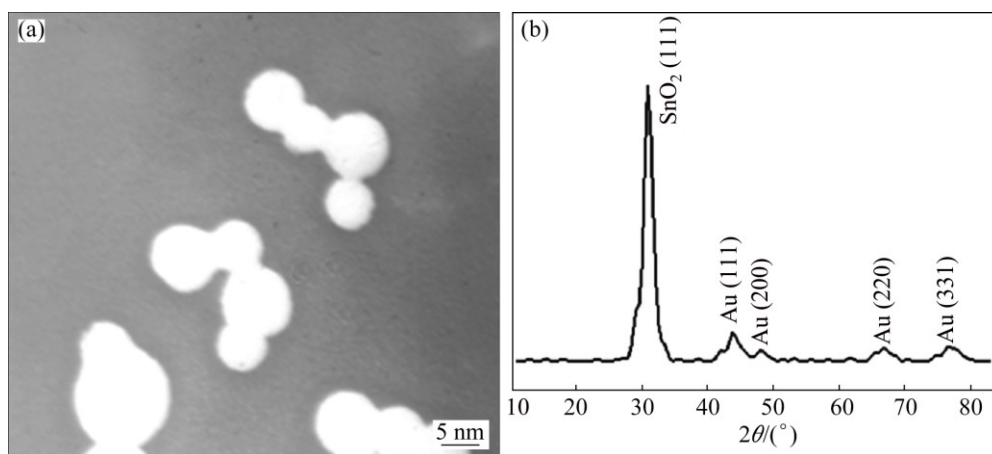


Fig. 2 TEM image (a) and XRD pattern (b) of GNPs

3–5 nm and a spherical shape for GNPs are evident.

The SnO_2 solution was mixed with GNP solution at different ratios and the results of the sensors were compared for different parameters. The amount of additive agent in the GNP- SnO_2 sensor was then optimized. Figure 3 shows the XRD patterns for SnO_2 and GNP- SnO_2 nanoparticles calcined at 600 °C for 2 h. When compared with the standard pattern of JCPDS (File No. 41–1445), the peaks agreed with the tetragonal structure of SnO_2 crystal. The XRD pattern of the GNP- SnO_2 showed the peak of GNP at about $2\theta=42^\circ$ (111), $2\theta=51^\circ$ (200), $2\theta=67^\circ$ (220) and $2\theta=75^\circ$ (331). The diffraction peaks of SnO_2 in the GNP- SnO_2 sample became sharper, indicating an increase in the size of the SnO_2 particles.

Figure 4 shows the SEM images of pure- SnO_2 and GNP- SnO_2 samples. The mean diameters of pure SnO_2 and GNP-doped SnO_2 were about 20 and 35 nm, respectively, which indicates that the average grain size increases with the addition of GNP. So, the addition of GNPs increases the grain boundaries and the electronic transmission channels, which are important for the current passing through the sensor.

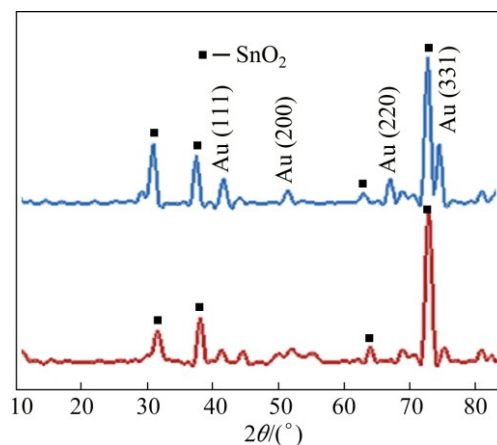


Fig. 3 XRD patterns of pure SnO_2 and GNP- SnO_2 calcined at 600 °C

To improve the selectivity and sensor response, catalyst materials such as noble metals can be added to SnO_2 solution. Au is one of the most interesting catalysts for CO gas sensing. In the present study, we produced five sample solutions containing different amounts of GNP solution ($m(\text{Au})/m(\text{Sn})=1.143\times 10^{-3}$ – 6.889×10^{-4}) and related sensors were tested under identical conditions

to obtain the best Au/Sn mass ratio. Figure 5 shows the response of the GNP-SnO₂ sensors at five GNP ratios and different temperatures. All samples were produced under the same conditions. The best performance for GNP-SnO₂ sensors was observed at 3.28×10^{-5} g GNP in 1 mL of SnO₂ solution ($m(\text{Au})/m(\text{Sn}) = 3.7663 \times 10^{-4}$) at a sintering temperature of 500 °C (Fig. 6). This ratio was then used for all further tests ($m(\text{Au})/m(\text{Sn}) = 3.7663 \times 10^{-4}$).

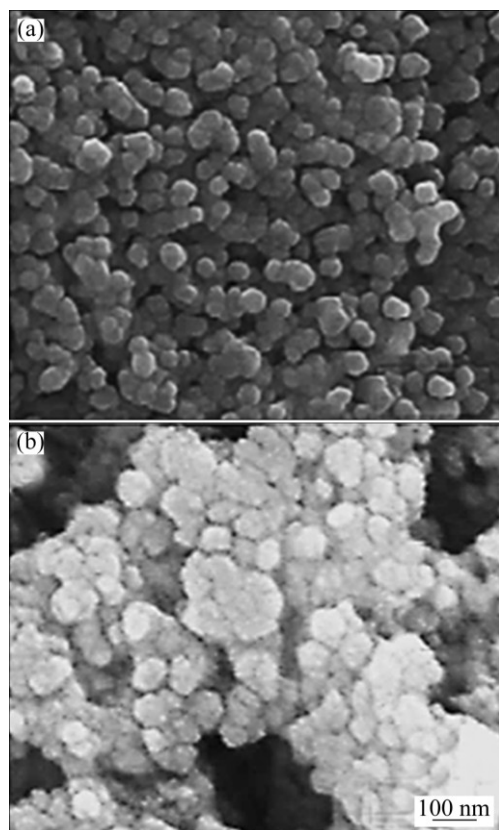


Fig. 4 SEM images of pure-SnO₂ (a) and GNP-SnO₂ (b)

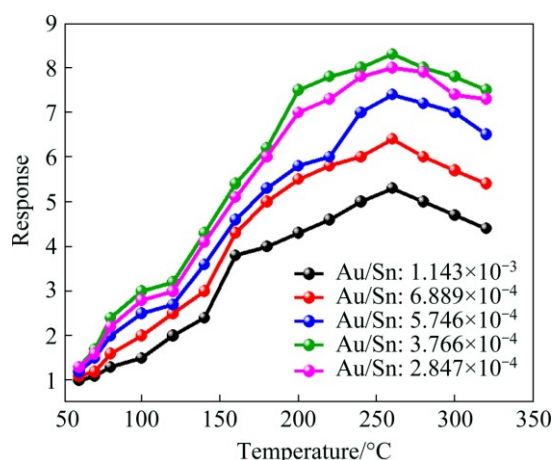


Fig. 5 Response of GNP-SnO₂-based sensor to 20×10^{-6} CO gas with different amounts of GNP as function of temperature

Testing was carried out at different temperatures to obtain the best operating temperature. The response

values for the pure SnO₂ sensor at different concentrations of CO gas are shown in Fig. 6(a). As the temperature increased, the response value increased. It continued up to about 340 °C, then the response gradually decreased. The optimal working temperature for the sensor was determined to be 340 °C for the pure SnO₂ CO gas sensor. The response ($R_{\text{air}}/R_{\text{gas}}$) for $(20-80) \times 10^{-6}$ CO gas was about 4 to 12.8. For GNP-doped samples, the operating temperature decreased to about 260 °C, and the response at this

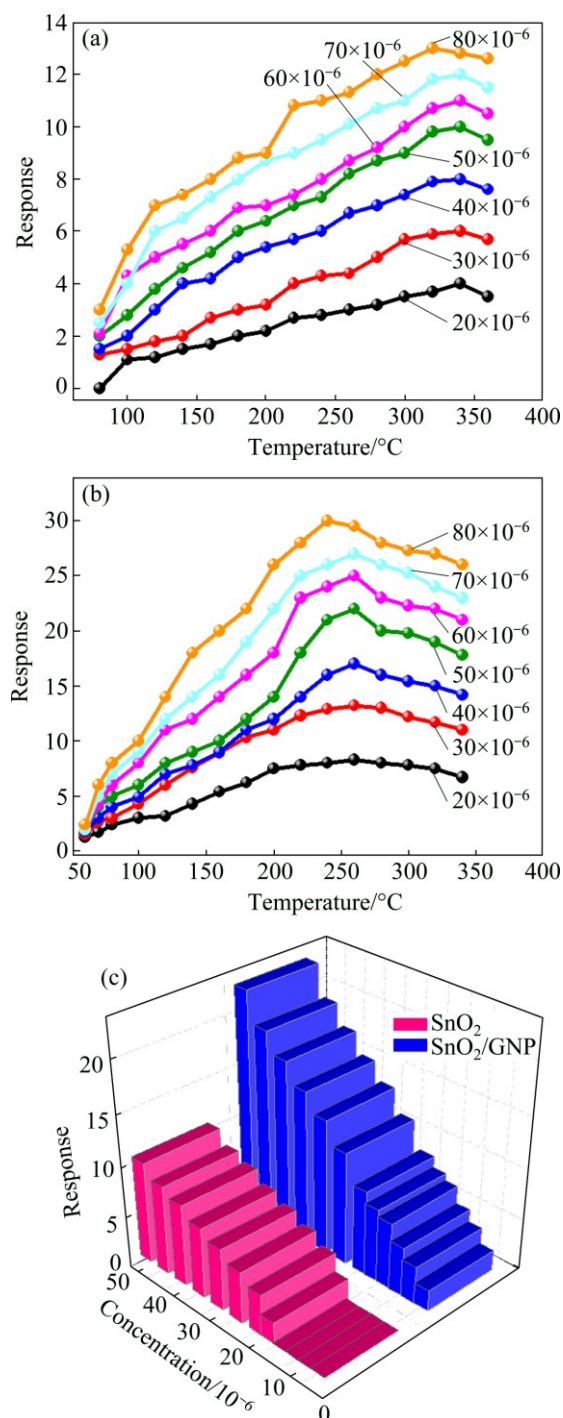


Fig. 6 Response of sensor at different CO concentrations as function of temperature: (a) pure-SnO₂; (b) GNP-SnO₂; (c) CO gas concentration threshold

temperature for $(20\text{--}80)\times 10^{-6}$ CO was about 8.3 to 29.5 (Fig. 6(b)).

For comparison, the response of GNP-SnO₂ samples produced at $\text{Au/Sn}=3.7663\times 10^{-4}$ is improved by 2-fold at different CO concentrations. Besides, the operating temperature for the doped sensor dropped to 260 °C, which was 80 °C lower than the pure sensor. This was the result of using gold catalyst to lower the activation energy required for the reactions to detect CO gas. Besides, since all tests were performed at a constant illumination of sunlight, the photo catalytic effect of the GNPs played an important role in lowering the operating temperature. UV radiation can reduce the operating temperature too. The photocatalytic effect of GNPs which tends to absorb UV-visible light illumination and gas sensing activation is due to their LSPR effect.

Figure 6(c) demonstrates and compares the outcomes for pure and optimized GNP-SnO₂ samples as a function of CO concentration. The CO gas concentration thresholds for pure SnO₂ and SnO₂/GNP samples were 5×10^{-6} and 20×10^{-6} , respectively. The pure SnO₂ sample showed no noticeable response at less than 18×10^{-6} CO.

Figure 7 shows the response and recovery time of pure-SnO₂ and GNP-SnO₂ sensors at 50×10^{-6} CO as a function of temperature. The response and recovery time for the pure SnO₂ sensor was about 10 and 14 s at 340 °C, respectively. These values for the GNP-SnO₂ sensor decreased to about 4 and 6 s at 260 °C, respectively.

YANAGIMOTO et al [37] made Au/SnO₂ core-shell nanoparticle CO sensors using the precipitation (A) and microwave hydrothermal synthesis (B) methods. The sensors were tested in the presence of 1000×10^{-6} CO gas at 300 °C. This sensor recorded responses of about 0.18 and 0.965 for A and B, respectively. The response time for sensors A and B was about 1.13 and 0.57 min, respectively. The recovery time was also about 6.7 and 15 min for sensors A and B, respectively. In all sensing specifications, our sensors showed higher performances which include lower operating temperature by 40 °C and higher responses at lower gas concentrations (at 50×10^{-6} CO gas, the response was about 22). Besides, the response and recovery time of our sensor was about 4 and 6 s, respectively, which is tremendously lower than that obtained by YANAGIMOTO et al [37].

Figures 8(a) and (b) show the transient response of pure-SnO₂ and GNP-SnO₂ sensors for 50×10^{-6} CO gas. Figures 8(c) and (d) show the transient response for 20×10^{-6} to 60×10^{-6} CO gas as a function of time at their optimal operating temperatures.

Figure 9 shows the response of the SnO₂ and GNP-SnO₂ sensors to several 50×10^{-6} gases at their optimal operation temperatures. The GNP-SnO₂ sensor showed the lowest response to NH₃ gas of 12 and the

highest response for CO of about 22. The pure SnO₂ sensor showed the lowest response of 5 to NH₃ and the highest response of about 12 for H₂. The GNP-SnO₂ sensor responded well to CO, suggesting that it can be employed to detect this gas for different usages.

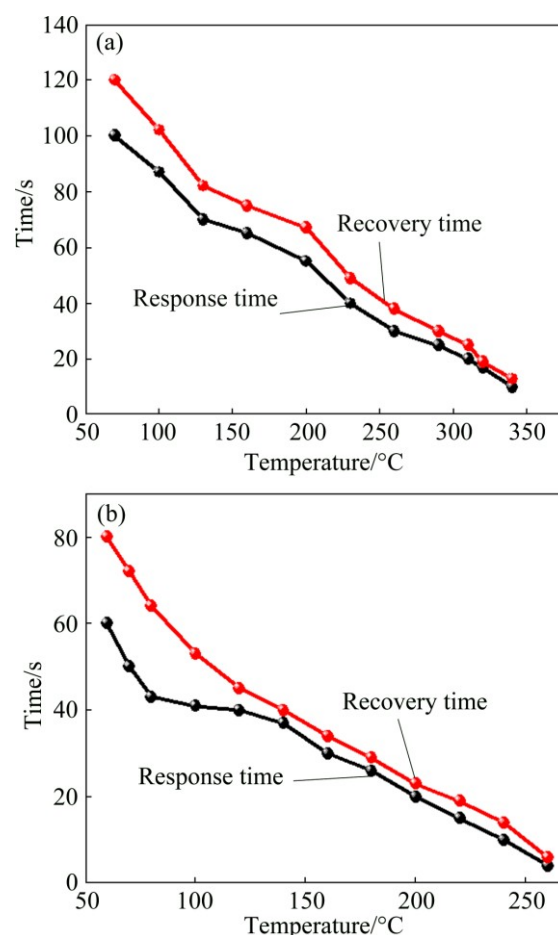


Fig. 7 Response and recovery time of sensor for 50×10^{-6} CO as function of temperature: (a) Pure-SnO₂; (b) GNP-SnO₂

4 Gas-sensing mechanism

The detection mechanism of our sensors in the presence of a reducing gas such as CO involves the partial chemical reduction of the sensitive layer surface [13]. At higher temperatures, the O₂ molecules ionized on the SnO₂ nanoparticles to form active ionic oxygen species like [O⁻]. Due to the high electron affinity of oxygen and because metal oxide semiconductors are *n*-type, they extract electrons from the semiconductor conduction band and create a depletion area on the surface of the grains, decreasing the effective radius of the grains for electron transport. By introducing CO gas, a reaction between the ionic oxygen atoms [O⁻] and CO molecules occurs [$\text{CO} + \text{O}^- \rightarrow \text{CO}_2 + \text{e}^-$]. This interaction releases electrons and re-injects them into the depletion region created in SnO₂ nanoparticles surfaces. This significantly reduces the height of barriers created

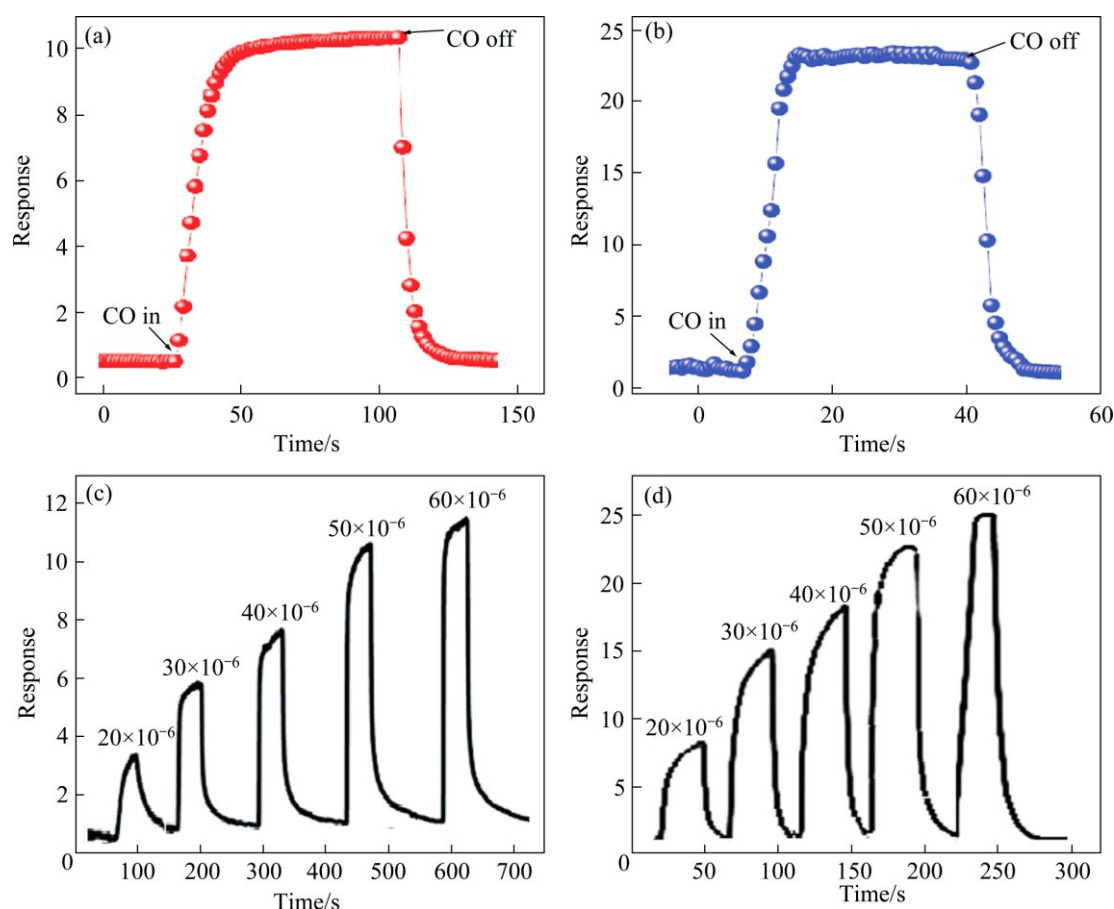


Fig. 8 Transient response of sensors at optimal temperature: (a) Pure-SnO₂ at 50 × 10⁻⁶ CO; (b) GNP-SnO₂ at 50 × 10⁻⁶ CO; (c) Pure-SnO₂ at (20–60) × 10⁻⁶ CO; (d) GNP-SnO₂ at (20–60) × 10⁻⁶ CO

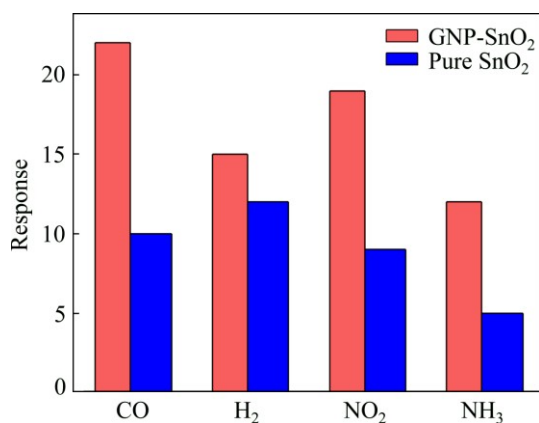


Fig. 9 Response of sensors at different 50 × 10⁻⁶ gases

between neighboring particles, resulting in an increase in sensor conductance. GNPs also cover SnO₂ nanoparticles to some extent but CO molecules can penetrate through the free spaces between the particles and react with ionic oxygen species (O₂⁻, O₂²⁻, O⁻) [14,21,25].

Through reducing the activation energy required for interactions as mentioned, GNPs increase the sensor response. Besides, as GNPs contact with SnO₂ nanoparticles, a Schottky barrier forms between them and electrons flow from the SnO₂ nanoparticles to the

GNPs. This occurs because of the work function difference between the gold and SnO₂ nanoparticles. This widens the depletion region into the SnO₂ nanoparticles and tends to further reduce the sensor conductivity. So, when CO molecules interact with pre-adsorbed oxygen ionic atoms to release and re-inject electrons into the depletion region created in SnO₂ nanoparticles, the changes of the sensor conductivity will be improved [14,21,25]. The GNPs affect the rate of attraction and repulsion of the gas molecules and improves the response and recovery time. These improvements are dependent on the interaction of the CO molecules and ionic oxygen species (O₂⁻, O₂²⁻, O⁻) under the catalytic effect of the GNPs [21,38,39].

5 Conclusions

The amount of GNPs optimized was used to create high performance GNP-SnO₂ sensors (Au/Sn = 3.7663 × 10⁻⁴). The optimal operating temperature is about 260 °C and the responses at concentrations of (20–80) × 10⁻⁶ are 8.3–29.5. This means that the operating temperature decreases by about 80 °C and the response improved by more than 2-fold compared with

the pure samples. At 50×10^{-6} CO gas, the response and recovery time for the pure SnO₂ sensor were approximately 10 and 14 s, and for the GNP-SnO₂ sensor, these values were approximately 4 and 6 s, respectively. This means that the sensor speed increases by more than 2-fold. The selectivity of the sensor response was evaluated for several gases. The GNP-SnO₂ sensor shows the best response for CO, suggesting that it can be employed for different related usages.

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GNP-SnO₂ 纳米气敏元件的气敏性能

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摘 要: 合成了不同金属纳米颗粒含量的 SnO₂ 纳米颗粒, 并研究了其 CO 气敏性能。采用溶胶-凝胶法制备初始溶液, 通过 SEM、TEM、XRD、DLS 和分光光度法表征纳米颗粒。在 340 °C 操作温度下, 纯 SnO₂ 纳米气敏元件对 $(20-80) \times 10^{-6}$ CO 的响应值为 4~12.8; 在 50×10^{-6} CO 浓度下, 其响应和恢复时间分别为 10 和 14 s。当 $m(\text{Au})/m(\text{Sn})=3.7663 \times 10^{-4}$ 时, GNP-SnO₂ 气敏元件具有良好的性能, 在 260 °C 优化操作温度下, 对 $(20-80) \times 10^{-6}$ CO 的响应值为 8.3~29.5。

关键词: GNP-SnO₂; SnO₂; 纳米颗粒; 气敏元件; 溶胶-凝胶法

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