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Influences of Impurity Gases in Air on Room-Temperature Hydrogen-Sensitive Pt–SnO_2 Composite Nanoceramics: A Case Study of H_2S

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Abstract: The slight but cumulative influence of impurity gases in air poses a great threat to the long-term stability of room-temperature gas sensors. Room-temperature hydrogen-sensitive Pt–SnO_2 composite nanoceramics of 5 wt% Pt were prepared through pressing and sintering. The response of a sample was over 10,000 after being exposed to 500 ppm H_2S–20% O_2–N_2 at room temperature, and the room-temperature hydrogen sensing capacity was seriously degraded even for samples that had aged dozens of days since H_2S exposure. Mild heat treatments such as 160 °C for 10 min were found able to fully activate those H_2S-exposed samples. As the peak of S 2p electron was clearly detected in H_2S-exposed samples, it was proposed that for room-temperature hydrogen-sensitive Pt–SnO_2 composite nanoceramics, H_2S exposure induced degradation results from the poisoning of Pt by H_2S deposited on it, which can be removed through a mild heat treatment. Periodic mild heat treatment should be a convenient and effective measure for room-temperature metal oxide gas sensors to achieve long-term stability through preventing the accumulation of impurity gases in air deposited on them.

Keywords: SnO_2; gas sensor; H_2S; catalyst; poisoning

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1. Introduction

Gas sensors are widely used in our daily life, from monitoring gaseous pollutants in air to alarming people to the presence of toxic gases (e.g., CO) [1–3] and detecting the leakage of explosive gases (e.g., H_2) [4–8]. Among the numerous gas sensors that have been successfully commercialized, those based on SnO_2 thick films are especially appealing for many applications due to their advantages of simple structure, easy operation, and low cost [9,10]. However, it has to be pointed out that these semiconductor gas sensors all have to work at elevated temperatures of around 400 °C [10–12], which not only increases power consumption but also brings a risk in itself, especially in the presence of explosive gases. Over recent decades, room-temperature semiconductor gas sensors have been intensively pursued by many researchers. Various kinds of low-dimensional metal oxide nanomaterials have been synthesized, and some of them have been revealed to exhibit impressive room-temperature gas sensing capabilities [13,14]. In particular, extraordinary room-temperature gas sensing capabilities have recently been observed in some bulk composites of noble metals and metal oxides that have been prepared through traditional pressing and sintering [15–17]. Compared with those low-dimensional metal oxide nanomaterials, these bulk composites possess such striking advantages as mass production and mechanical robustness for practical room-temperature gas sensing applications.

While metal oxide room-temperature gas sensors are becoming more and more promising for practical applications, some important but long neglected aspects have begun to be investigated for them. For example, high moisture resistance is important for gas sensors, and the moisture resistances of room-temperature hydrogen sensors based on Pt–SnO_2 and Pt–WO_3 composite ceramics have been systematically investigated recently. It is highly encouraging to see that through adopting SnO_2 agglomerate powder and WO_3 ultra-large grains for Pt–SnO_2 and Pt–WO_3 composite ceramics, respectively, their moisture resistances are dramatically improved while their high room-temperature hydrogen sensitivities remain quite unchanged [18,19]. Another encouraging result has been obtained for Pt–SnO_2 room-temperature hydrogen sensors and Pt–SnO_2 room-temperature CO sensors. Though their room-temperature gas sensing characteristics were found to degrade very seriously with time, a rather mild heat treatment, such as one at 150 °C for 15 min, was able to mostly recover their room-temperature gas sensing characteristics [20,21]. Obviously, such a finding on aging and activation should be of great importance for the commercialization of these metal oxide room-temperature gas sensors.

It is well known that air is a mixture of many gases. In addition to oxygen, nitrogen, and water vapor, many other gases of small amounts are also present, which are usually referred to as impurity gases in air. Some of these impurity gases are actually very reactive and are not only harmful to human health but may also show some interference to gas sensors exposed to air. To date, however, there have been no investigations on any impurity gases in air with regards to their influences on room-temperature metal oxide gas sensors, which will raise much concern over their intended applications. Given that H_2S is a rather common toxic pollutant in air [22–25], presently, we have studied its effects on room-temperature hydrogen-sensitive Pt–SnO_2 composite nanoceramics. With Pt as a highly stable and catalytic noble metal and SnO_2 as the most important metal oxide semiconductor for gas sensing, Pt–SnO_2 composite nanoceramics have been revealed to be highly promising for practical room-temperature hydrogen sensing applications [18,20]. The composite nanoceramics were found to show strong responses to H_2S at room temperature, and their hydrogen sensing characteristics were seriously degraded after the exposure. Fortunately, the degradation could be completely removed through a rather mild heat treatment (i.e., 160 °C for 10 min) and some interesting findings have been obtained for the degradation process. These results could be important for us to achieve a specific understanding of the influences of impurity gases in air on room-temperature metal oxide gas sensors and to take necessary measures to make the intended applications of room-temperature metal oxide gas sensors more reliable.

2. Materials and Methods

SnO_2 nanoparticles (50–70 nm) and Pt powder (<1 µm), both from Shanghai Aladdin, were dispersed into deionized water at a mass ratio of 95:5. After four hours of magnetic stirring, the suspension was dried at 120 °C for 4 h. The dried powder was pressed into pellets of 10 mm in diameter and 1 mm in thickness under 3 MPa. According to some previous investigations, the pellets were sintered at 825 °C for 2 h in air. A pair of rectangular gold electrodes in parallel (5 mm × 2 mm, with a 2 mm gap) were formed on a major surface of a sintered pellet through DC magnetron sputtering.

The gas control and resistance measurements were performed with a commercial gas sensing system (GRMS-215, Partulab Com., Wuhan, China) as shown in Figure 1. It was equipped with a cavity of approximately 350 mL within which the sensor under test was placed. For the response phase, gases of O_2, N_2, and 1.5% H_2–N_2/0.1% H_2S–N_2 were flown into the cavity through three gas flow valves at some designated ratios. The total gas flow rate was 300 mL/min. For the recovery phase, ambient air was pumped into the cavity at a rate of 10 L/min. A voltage of 10 V was applied between the two gold electrodes of the sample under test, and the change in resistance was automatically recorded by an electric meter (Keithley 2400 source). The change in the resistance of the sensor under different atmospheres was recorded automatically. During the whole experiment, the ambient temperature was kept at 25 °C and the relative humidity of air was kept at 40%.

Figure 1. Photograph of the commercial gas sensing system (GRMS-215, Partulab Com., Wuhan, China) used in this study.

X-ray diffraction (XRD) spectra were recorded on an X-ray diffractometer (SmartLab, Japan Rigaku) using Cu–Kα radiation. An X-ray photoelectron spectroscopy (XPS) analysis was performed at room temperature using a Thermo ESCALAB 250xi instrument. Electron binding energies were calibrated using the reference peak of C 1 s (284.6 eV). The microstructure was analyzed on a field emission scanning electron microscope (FESEM; SIGMA, ZEISS Corporation, Jena, Germany), and an energy-dispersive spectroscopy (EDS) analysis was performed with an OXFORD Aztec 250 instrument.

3. Results and Discussion

Pt–SnO_2 composite nanoceramics with a series of Pt contents have been prepared and investigated for room-temperature hydrogen sensing in some previous investigations [26]. Although highly impressive room-temperature hydrogen sensing characteristics can be observed for them over a rather large Pt content range, those samples of relatively high Pt contents have a striking advantage in long-term stability. For this reason, we chose a relatively high Pt content of 5 wt% for the samples to be further studied. Figure 2 shows the XRD pattern taken on the surface of a sample sintered at 825 °C. For this Pt content, three peaks from metallic Pt (JCPDS 04-0802) could be clearly detected. All other peaks could be clearly identified as those of rutile SnO_2 (JCPDS 41-1445). Obviously, these samples were composites of rutile SnO_2 and metallic Pt.

The microstructure of Pt–SnO_2 composite nanoceramics has also been revealed in some previous investigations. Generally speaking, there is no considerable shrinking for composite nanoceramics even after sintering at 1200 °C [17], and there is no substantial SnO_2 grain growth either. As shown in Figure 3a, which was taken for a fractured surface of a sample prepared in this study, numerous grains around 70 nm could be observed, and they were SnO_2 nano-grains according to the EDS analysis shown in Figure 3b. On the other hand, two much larger grains, around 250 nm in size, could be observed and identified as Pt grains. Obviously, such large Pt grains were from the as-received Pt powder, which was marked as <1 µm. As a matter of fact, according to a very recent paper, these large Pt grains are helpful for the samples to remain room-temperature hydrogen-sensitive over a long period of time [21].

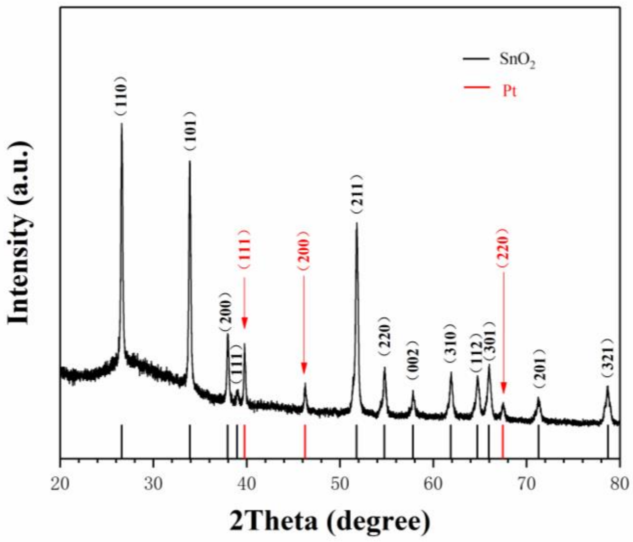

Figure 2. X-ray diffraction pattern taken on the surface of a pellet which was prepared using Pt powder (5 wt%) and SnO_2 nanoparticles and sintered at 825 °C in air.

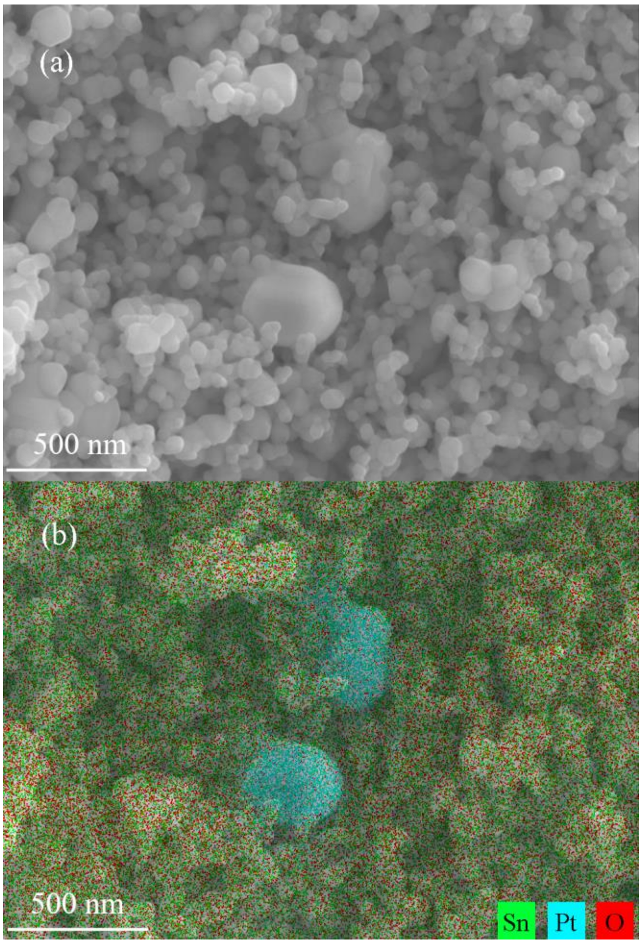

Figure 3. (**a**) SEM micrograph taken on a fractured surface and (**b**) SEM micrograph with EDS analysis for a pellet of 5 wt% Pt sintered at 825 °C for 2 h in air.

Highly impressive room-temperature hydrogen sensing characteristics have been obtained for Pt–SnO_2 composite nanoceramics in previous investigations. For the samples prepared in this study, their room-temperature hydrogen sensing characteristics were also very attractive, as shown by those obtained for a sample in Figure 4. The response of a sensor is usually defined as $S = Ra/Rg$, where Ra and Rg are the resistance values of the sensor in air and in test gas, respectively. As can be seen from Figure 3, the sample had responses of over 5000, 2000, 500, and 100 for 1%, 0.5%, 0.125%, and 0.0625% H_2–20%O_2–N_2, respectively. Moreover, the sample exhibited a fast response speed during every response, reaching a plateau in the resistance response in about 50 s. When exposed to air, the sample also returned to its initial resistance within a recovery time of 200 s. For 1% H_2–20% O_2–N_2, the sample's response and recovery times were 7 s and 150 s, respectively. Such room-temperature hydrogen sensing characteristics could be very appealing for practical applications.

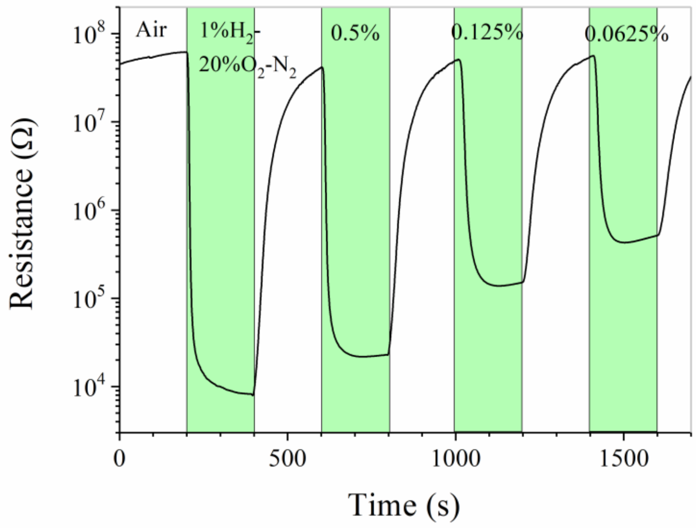

Figure 4. Resistance responses of a sample of 5 wt% Pt sintered at 825 °C to a series of concentrations of H_2 in 20% O_2–N_2 at room temperature (25 °C), and recovery in air of 40% RH.

In some previous investigations, the influences of water vapor on room-temperature hydrogen-sensitive metal oxides have drawn much attention and have been well explored [18,19]. As a result, some effective measures to minimize the influences of water vapor have been suggested. In contrast, the influences of impurity gases in air have not been investigated for any room-temperature gas-sensitive metal oxides to date. Given that H_2S is an important pollutant in air, we have chosen H_2S as an example of an impurity gas in air to study its influence on the room-temperature hydrogen-sensitive Pt–SnO_2 composite nanoceramics prepared in this study. First, some samples were exposed to H_2S at several concentrations, which were found to have surprisingly strong responses to H_2S at room temperature. When a sample was exposed to 500 ppm H_2S–20% O_2–N_2 at room temperature, its response was over 10,000, as shown in Figure 5. For reference, these samples usually have a response of around 100 for 625 ppm H_2–20%O_2–N_2 at room temperature. Obviously, these samples showed much stronger responses to H_2S than to H_2 at the same concentration at room temperature. However, their resistance could not be recovered in a reasonable time when air was introduced. For the sample shown in Figure 5, after being exposed to 500 ppm H_2S–20% O_2–N_2 for 200 s, its decreased resistance was only recovered by ~10 after 200 s in air. Similar results can be observed for samples exposed to H_2S at other concentrations. As their resistance cannot be fully recovered in air in a reasonable time, it can first be concluded that the samples prepared in this study are not suitable for room-temperature H_2S sensing.

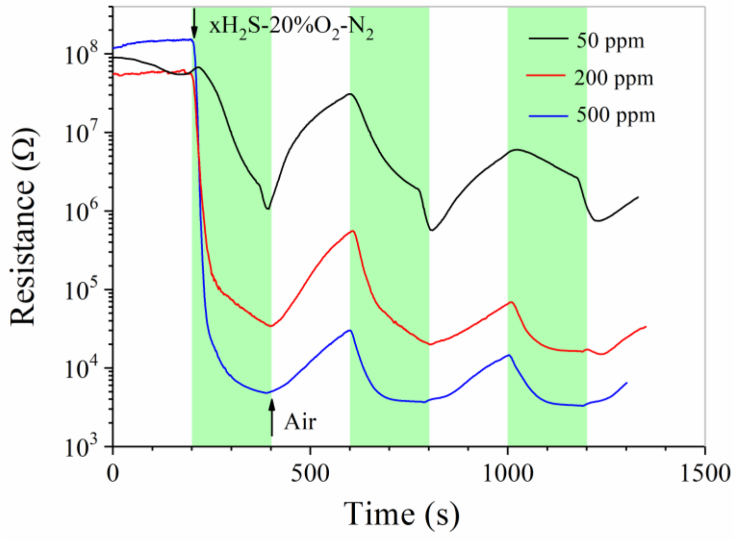

Figure 5. Responses to H_2S at room temperature for three Pt–SnO_2 composite nanoceramic samples.

Since these samples showed such strong responses to H_2S at room temperature, the influence of H_2S as an impurity gas in air on their intended room-temperature hydrogen sensing had to be seriously studied. For this purpose, a representative sample was exposed to 25 ppm H_2S–20% O_2–N_2 at room temperature for 600 s and then kept in air at room temperature for a long period of time. Its room temperature responses to hydrogen were measured repeatedly after some time intervals, and those obtained after 7 days and 30 days are shown in Figure 6. It can be clearly seen that although the sample had mostly recovered its resistance in air, its room-temperature hydrogen sensing characteristics were still seriously degraded even after 7 and 30 days had passed since the H_2S exposure separately. While the response to 1% H_2–20% O_2–N_2 measured after 30 days was much stronger than that after 7 days, its recovery in air was still very slow. As a matter of fact, for samples aged in air for some long periods of time, their recovery speeds are usually the most seriously degraded, as shown by that obtained for a three-month-aged sample in Figure 6. It should be pointed out that this H_2S-exposed sample showed much more serious degradation than the three-month-aged sample, which indicates that the degradation observed for it had mostly resulted from the H_2S exposure. Obviously, H_2S exposure has a serious influence on room-temperature hydrogen sensors based on Pt–SnO_2 composite nanoceramics, and the influence is difficult to eliminate through an aging process at room temperature.

For room-temperature hydrogen-sensitive Pt–SnO_2 composite nanoceramic samples, such a serious effect of H_2S exposure should be taken into full consideration in their future applications. On the one hand, their application should be avoided in environments where H_2S at considerable concentrations is present. On the other hand, given that H_2S is a common impurity gas in air, there exists a high possibility that H_2S at low concentrations in air has a slight negative influence on hydrogen sensors based on Pt–SnO_2 composite nanoceramics, which does not diminish but accumulates with time at room temperature. In this way, the room-temperature hydrogen sensing characteristics of Pt–SnO_2 composite nanoceramics will degrade gradually with time. It is therefore necessary to find a simple and effective method to prevent the accumulation of H_2S exposure influence on Pt–SnO_2 composite nanoceramics. We tried to recover H_2S-exposed samples through some mild heat treatments, and the results were very encouraging. As shown in Figure 7, a Pt–SnO_2 composite nanoceramic sample was greatly degraded in its room-temperature hydrogen sensing performance after it was exposed to 25 ppm H_2S–20% O_2–N_2 for 600 s at room temperature, while its room-temperature hydrogen sensing performance was completely restored through a heat treatment of 160 °C for 10 min in air. It indicates that although the influence of H_2S exposure cannot be eliminated through a room-temperature aging process, it can be readily eliminated through a rather mild heat treatment. Obviously, the influence of H_2S exposure on Pt–SnO_2 composite nanoceramics is sensitive to temperature, and a mild heat treatment will be adequate for Pt–SnO_2 composite nanoceramic samples to remain room-temperature hydrogen-sensitive against the influence of H_2S as an impurity gas in air.

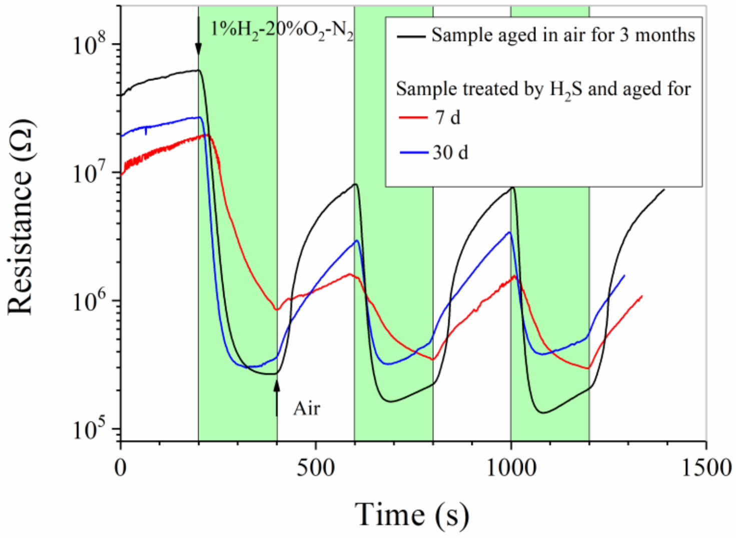

Figure 6. Room-temperature responses to 1% H_2–20% O_2–N_2 and recoveries in air for two Pt–SnO_2 composite nanoceramic samples: one had been aged in air for three months, and the other one had been exposed to H_2S at room temperature and then aged in air for 7 d and 30 d separately.

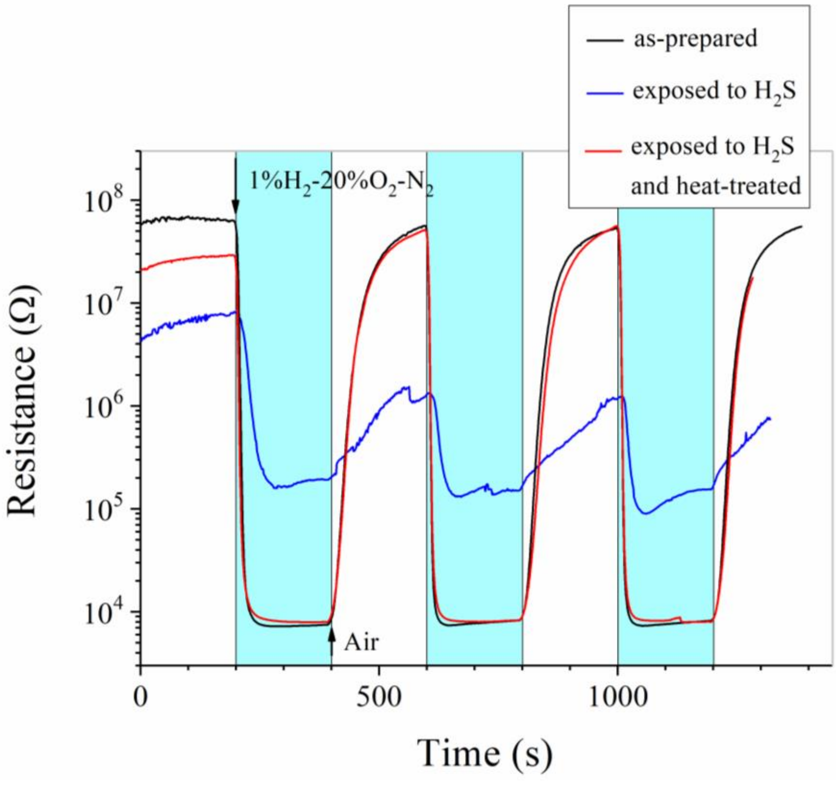

Figure 7. Room-temperature responses to 1% H_2–20% O_2–N_2 and recovery in air for a Pt–SnO_2 nanoceramic sample: as-prepared, being exposed to H_2S, and being exposed to H_2S and then being heat-treated, separately.

We further investigated the influence of H_2S exposure on Pt–SnO_2 composite nanoceramics through XPS analyses. Figure 8 shows some XPS spectra obtained for a sample after being exposed to 25 ppm H_2S–20% O_2–N_2 at room temperature for 600 s, and then after being heat-treated at 160 °C for 10 min in air, separately. As shown in Figure 8a, the peak of the S 2p electron could be clearly seen after the sample's exposure to H_2S, which indicates that some H_2S must have been absorbed by the sample, and its room-temperature hydrogen sensing behavior was thus affected. On the other hand, as shown in Figure 8b, the peak of the S 2p electron could no longer be observed after the sample was heat-treated at 160 °C for 10 min in air. Together with the activation observed with the heat treatment shown in Figure 7, it is reasonable to assume that such a heat treatment is able to activate H_2S-influenced samples by removing H_2S from them. As a matter of fact, Pt plays a vital catalytic role in achieving the room-temperature hydrogen sensing capability in Pt–SnO_2 composite nanoceramics. Meanwhile, for catalysts, there exists a common phenomenon known as poisoning in which the active sites of the catalysts are blocked by some molecules deposited on them, and their catalytic effects are greatly degraded [27–29]. Obviously, the influence of H_2S exposure observed in this study can be well explained in terms of Pt poisoning by the H_2S deposited on it, and in turn, the mild heat-treatment-induced activation can be explained in terms of H_2S desorption from Pt.

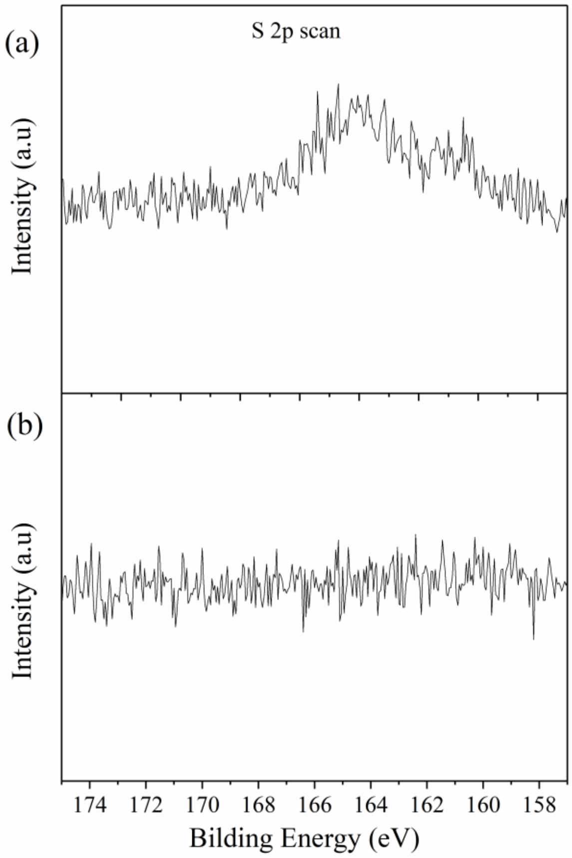

Figure 8. XPS spectra of S 2p electron in (**a**) a sample of Pt–SnO_2 nanoceramic exposed to H_2S at room temperature and (**b**) a sample of Pt–SnO_2 nanoceramic heat-treated at 160 °C for 10 min in air after H_2S exposure.

As for poisoning by toxic gases, not only the concentration of the gases but also the duration of exposure to the gases has an important influence on the degree of poisoning. It is well known that a complete recovery is easier to achieve from slight poisoning than from serious poisoning. It suggests that if Pt–SnO_2 composite nanoceramics can be periodically heat-treated, the concentration of H_2S deposited on them can be kept under a very low level. The degree of poisoning by H_2S can thus be minimized, which is important not only for maintaining extraordinary room-temperature hydrogen sensing characteristics but also for a complete recovery through mild heat treatment. Further investigations on such periodic heat treatments for Pt–SnO_2 composite nanoceramics are being conducted.

There are numerous impurity gases in air, and other impurity gases should also be carefully investigated with regard to their influence on room-temperature metal oxide gas sensors. As a matter of fact, obvious aging has recently been observed for Pt–SnO_2 composite nanoceramics exposed in air, which is also mostly characterized by decreases in recovery speeds following the degradation induced by H_2S exposure [20]. On the other hand, it is more favorable for impurity gases to deposit on metal oxides at room temperature than at elevated temperatures. It is thus reasonable to assume that the deposition of impurity gases such as H_2S in air must have played a vital role in the observed aging at room temperature. Given the stability of Pt, most impurity gases should be able to be completely removed from Pt through some mild heat treatments, especially when the concentration of the gases is very low, and the room-temperature hydrogen-sensitive capability of Pt–SnO_2 composite nanoceramics is thus fully activated. Periodic mild heat treatment is therefore a convenient but effective measure for Pt–SnO_2 composite nanoceramics to remain room-temperature hydrogen-sensitive in air over long periods of time.

4. Conclusions

Pt–SnO_2 composite nanoceramics with an extraordinary room-temperature hydrogen sensing capacity were prepared through pressing and sintering. The samples were found to show extremely strong responses to H_2S at room temperature, and their room-temperature hydrogen sensing capacity was seriously degraded even after they had aged for dozens of days since H_2S exposure. Fortunately, the properties of those H_2S-exposed samples were fully restored through rather mild heat treatments, such as one at 160 °C for 10 min in air. XPS analyses showed the presence of sulfur in H_2S-exposed samples, and it was removed through mild heat treatments. These results suggest that the Pt in the Pt–SnO_2 composite nanoceramics must have been poisoned by the H_2S deposited on it, and it could be activated when H_2S was removed through heat treatments. Together with recent findings on room-temperature aging for Pt–SnO_2 composite nanoceramics, it can be concluded that the influences caused by the deposition of H_2S and other impurity gases in air can also be eliminated through a mild heat treatment. Periodic mild heat treatment should be indispensable and effective for Pt–SnO_2 composite nanoceramics to be room-temperature hydrogen-sensitive over long periods of time in air.

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