



The superior performance of the electrochemically grown ZnO thin films as methane sensor

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ARTICLE INFO

Article history:

Received 13 December 2007

Received in revised form 22 February 2008

Accepted 25 February 2008

Available online 29 February 2008

Keywords:

Nanoporous ZnO

Electrochemical anodization

MIM configuration

Methane sensing

Pd–Ag (26%) and Rh catalytic metal contacts

ABSTRACT

Pd–Ag/ZnO/Zn and Rh/ZnO/Zn MIM (metal–insulator–metal) gas sensors were fabricated using nanoporous ZnO thin films, obtained by an electrochemical deposition method in the absence and presence of UV light. A high-purity Zn anode, a Pt cathode, a calomel reference electrode and a 0.3-M oxalic acid electrolyte were used for deposition. Pd–Ag (26%) and Rh were used separately as the catalytic metal electrodes to fabricate the two different types of MIM configurations. A gas response of the order of 3.85 ± 2 , a response time of 5 ± 0.5 s and a recovery time of 16 ± 0.5 s were obtained with the Pd–Ag contact, while the Rh contact showed a response of the order of 4.82 ± 2 , a response time of 24 ± 0.5 s and a recovery time of 72 ± 0.5 s, at the optimum temperature of 220°C , which is the lowest temperature so far reported for metal oxide sensors to sense 1% methane in a N_2 carrier gas. The undoped zinc oxide thin films grown by UV-assisted electrochemical anodization of high-purity Zn demonstrated a better performance for methane sensing. The experiments were repeated in synthetic air and a somewhat reduced performance was observed. The selectivity in the presence of hydrogen and the stability of the sensors were studied.

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1. Introduction

A nanocrystalline-nanoporous ZnO thin film is one of the most promising materials for chemical gas sensors due to its large surface to volume ratio, minimum preparation cost and high compatibility with Si microelectronic processing [1–3]. The principle of gas sensing relies on the change in resistivity on exposure to target gases. The gas sensing can be improved by the incorporation of a noble metal catalyst to the basic sensing material. SnO_2 -based gas sensors in the form of thick films, porous pellets or thin films along with Pt or Pd as a catalyst are widely used for monitoring of explosive and toxic gases in industry, urban and domestic life. Other oxides (Ga_2O_3 , TiO_2 , WO_3 , etc.) [4–8] have also been investigated for their gas sensing properties. Nowadays challenges are made to attain high gas response to lower ppm of testing gases, short response and recovery times, selectivity and long-range stability of the gas sensors. Nanocrystalline metal oxide films demonstrate remarkable gas sensing properties when the crystallite size becomes comparable to Debye length. ZnO films can be prepared by the methods such as sputtering, pulsed laser deposition [9], chemical vapor deposition [10], sol–gel processing and electrochemical processing [11–12].

The functional characteristics of gas sensors, e.g. response, response time and recovery time have been improved over the resistive Taguchi-type sensors by adopting Schottky structures with catalytic metal contact. The vertical structure fabricated by growing metal oxides on the conducting surface and depositing a catalytic metal on the oxide improves the functional properties of the sensor devices because the electrons generated in the catalytic metal–oxide interface can be collected by the second electrode in the back with minimum carrier annihilation in the transport mechanism. This type of MIM configuration was first studied by Fonash et al. [13]. The basic principle of the current conduction mechanism of an MIM sensor configuration is that the electrons move from the upper metal electrode to the lower one through the active insulating layer vertically on the basis of back-to-back Schottky barrier junctions. In our MIM configuration, since the Zn contact to the ZnO layer is ohmic by nature, only the noble metal/ZnO junction acts as the barrier against the electron flow. A room temperature hydrogen sensor using ZnO MIM structure was first reported by Dutta and coworker [14]. Bhattacharyya et al. [15] reported a MIM methane sensor using sol–gel grown ZnO as an active insulator, Pd–Ag (catalyst) and Zn metal electrodes. In this paper we report on a comparative study of Pd–Ag (26%)/ZnO/Zn and Rh/ZnO/Zn MIM sensors for methane sensing using nanoporous ZnO thin films deposited by an electrochemical method both in the presence and in the absence of UV radiation.

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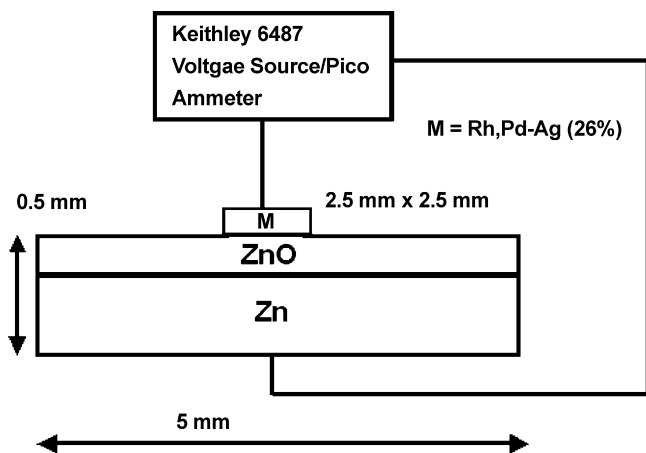


Fig. 1. The schematic of the MIM sensor structure.

2. Experimental

Nanoporous ZnO thin films were prepared by electrochemical anodization of high-purity Zn (5 mm × 5 mm) of thickness 0.5 mm at room temperature using a Pt counter electrode and a calomel reference electrode in 0.3 M oxalic acid as an electrolyte. A constant 10 V supply (Scanning Potentiostat-362, Princeton Applied Research, USA) was used during anodization and the reduction of current with the formation of insulating ZnO was recorded till the saturation of the current indicated the termination of the oxide formation. The anodization was carried out without and with UV radiation (Dr. Grobel, UV-Elektronik GmbH UV-LQ 400) incident on the anode in the pulse mode (10 min). X-ray diffraction (XRD) (Bruker DH Advance) and field emission scanning electron microscopy (FESEM) (JEOL, JSM-6700F) were employed to study the polycrystalline nature, crystal size, surface morphology and pore size of the deposited ZnO thin films. A Pd–Ag (26%) and Rh (2.5 mm × 2.5 mm) catalytic metal of thickness 0.2 μm was deposited on the ZnO films by e-beam evaporation using an Al metal mask. Electrical contacts were taken by using fine copper wire and silver paste. The thickness of the ZnO thin film grown in the absence and presence of UV light was 8.2 μm and 4.8 μm, respectively, as was measured by a surface profilometer (DEKTAk Surface profilers, Sloan Technology, Veeco, Version-1.05). The schematic of the MIM sensor structure is shown in Fig. 1. Rh/ZnO/Zn MIM structures with ZnO films grown in the absence and presence of UV radiation are designated as X₁ and X₂ sensors. Similarly Pd–Ag/ZnO/Zn MIM structures with ZnO films grown in the absence and presence of UV light are designated as X₃ and X₄ sensors in the subsequent

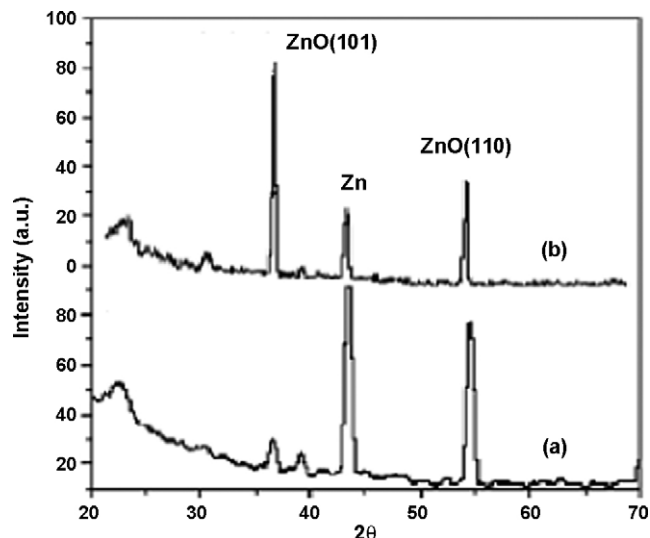


Fig. 2. XRD patterns of ZnO thin films obtained by anodic oxidation of Zn in 0.3 M oxalic acid in the (a) absence and (b) presence of UV light.

discussions. The gas sensor characteristics in the absence and presence of methane were studied inside a closed corning glass tube with inlet and outlet provisions for gases and the tube was placed coaxially inside a resistively heated furnace with a 4-cm constant temperature zone. The temperature was controlled within ±1 °C by a precise temperature controller with a built-in copper constantan thermocouple.

For sensor study high-purity methane, high-purity nitrogen and synthetic air were used. To measure and to control the flow rates of the gases precisely throughout the experiments, mass flow meters and controllers (Digiflow, USA) were used. The current–voltage characteristics of the sensors in the absence and presence of methane were studied by a Keithley Pico ammeter–voltage source (Model 6487).

3. Results and discussions

The XRD of the anodized ZnO thin films are shown in Fig. 2. It is observed that the films prepared with UV light have higher peak intensity of ZnO (1 0 1) than the films grown without UV light. The increase of intensity is due to the grain growth in the presence of UV light because of enhanced rate of oxidation by excess hole generation. The crystallite size was calculated from the Scherrer formula. FESEM pictures (Fig. 3) clearly show that the films grown with UV light has enlarged pore size as compared to the film grown with-

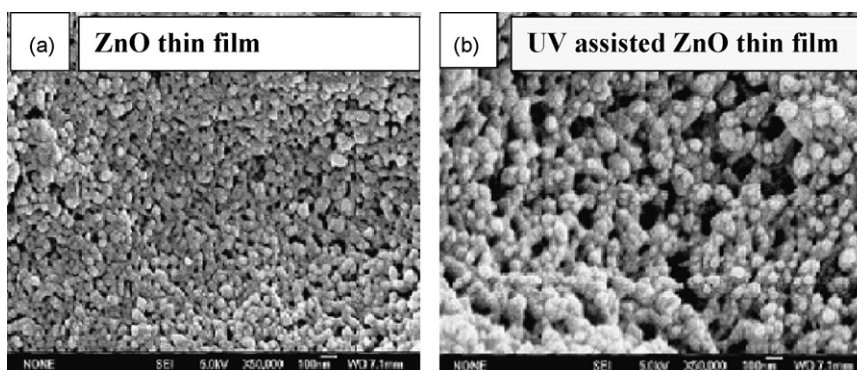


Fig. 3. FESEM images of ZnO thin films grown (a) without and (b) with UV light in 0.3 M oxalic acid. It is observed that the pore size and grain size of UV-assisted ZnO thin film increases as compared to the film grown without UV light (identical magnification (50,000×)).

Table 1
Effect of UV light on the crystal size, pore size and thickness of ZnO films

Parameters	Without UV light	With UV light
Grain size (nm)	2.41	2.67
Pore size (nm)	19–35	40–45
Thickness (μm)	8.2	4.8

out UV light. The average crystallite size, the pore diameter and the thickness are summarized in Table 1. The MIM sensors were studied with different methane concentrations (0.01%, 0.05%, 0.1%, 0.5% and 1%) in nitrogen and in synthetic air separately as carrier gases over the temperature range 200–250 °C. A Pd–Ag (26%) alloy was used as the metal contact instead of Pd. The pure Pd is a good catalyst for methane sensing, but there is a stability problem due to a phase transition and embrittlement because of palladium hydride formation. To avoid the instability of the sensor structure, Pd–Ag (26%) alloy was used as the catalytic contact for methane sensing [16]. The *V–I* characteristics of the MIM sensors were recorded at all the temperatures of measurements. The response (*S*) is defined here as the ratio of the change of forward current in the presence of methane to the current in air at constant voltage and it is expressed as

$$S = \left(\frac{I_g - I_a}{I_a} \right)_V \quad (1)$$

where *I_g* is the current in the presence of gas and *I_a* is the current in air.

The change of response of the MIM sensors with temperature for both type of contacts were investigated. Fig. 4 shows that the response is maximum at 220 °C for both sensor structures. The figure also reveals that X₁ and X₂ sensors show higher response than X₃ and X₄ sensors. It is observed from the response versus voltage curves for both types of contacts (Fig. 5) that the maximum response occurs at only 0.4 V for both sensors, which is very significant for low power consumption of the sensor devices. Fig. 6(a) and (b) show the response versus temperature curves for both MIM sensors to 1% CH₄ in pure N₂ and in synthetic air, respectively. The response of the sensors is higher in nitrogen than in synthetic air at 0.4 V and at 220 °C.

Fig. 7 shows the response versus temperature curves of X₁, X₂, X₃ and X₄ sensors to 1% hydrogen and 1% methane both in nitrogen. It is evident from the figure that hydrogen shows the maximum response at room temperature while methane exhibits the maximum response at 220 °C. Therefore, the selectivity of

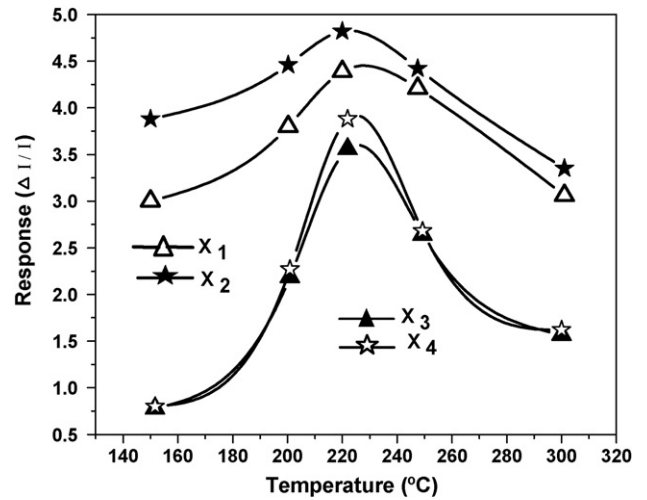


Fig. 4. Response vs. temperature plots of MIM sensors to 1% CH₄ in pure N₂ environment. The maximum response occurs at 220 °C. The response of Rh/ZnO/Zn sensors (X₁ and X₂) is higher than Pd–Ag/ZnO/Zn sensors (X₃ and X₄).

methane in the presence of hydrogen is improved at higher temperature. However, it is noted that for both Rh and Pd–Ag (26%) the response to methane was higher with X₂ and X₄ sensors. UV grown ZnO films showed larger pore size, thereby improving the gas adsorption–desorption kinetics and thus gas response characteristics. With UV grown ZnO the better sensor performance is also due to the lower thickness of the films, which lowers the series resistance of the sensor structures. The methane sensing response exhibited by the MIM sensors with ZnO films grown by the electrochemical anodization method is quite high as compared to that reported by the authors with sol–gel grown ZnO films [15] due to the improved nanocrystalline nature of ZnO. As the grain sizes of Rh and Pd–Ag (26%) alloy on ZnO vary in the range of 10–20 nm and 20–40 nm, respectively, the adsorption rate of oxygen on these catalytic metals should be high. At an elevated temperature, the catalytic metal atoms (Pd and Rh) are weakly bonded with oxygen molecules. The resulting complex dissociates and oxygen atoms (reactions (2) and (3)) are produced [17,18]. The oxygen atoms then undergo a spillover process and finally form negatively charged surface ions by gaining electrons from the surface of ZnO, yielding an electrostatic potential in the junction. The nature of the electrostatic potential is very high due to the small grain size (below

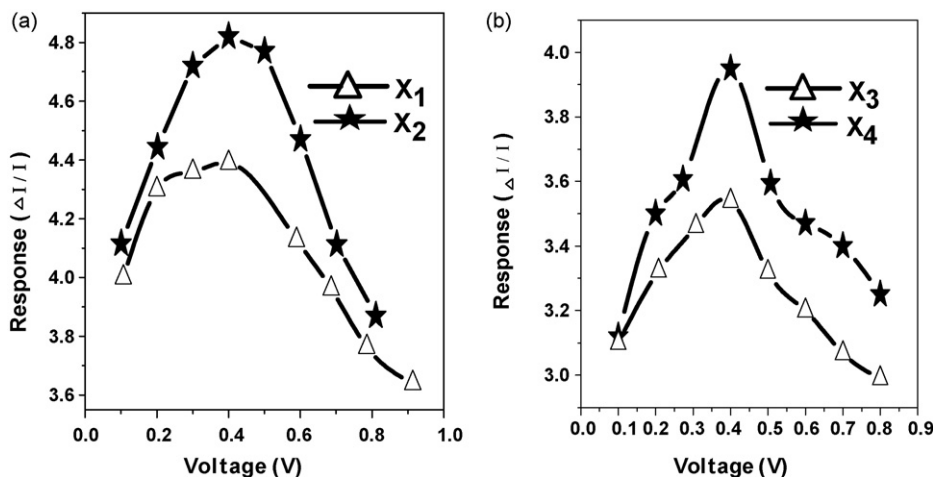


Fig. 5. Response vs. voltage curves of MIM sensors. The maximum response occurs at 0.4 V for both cases.

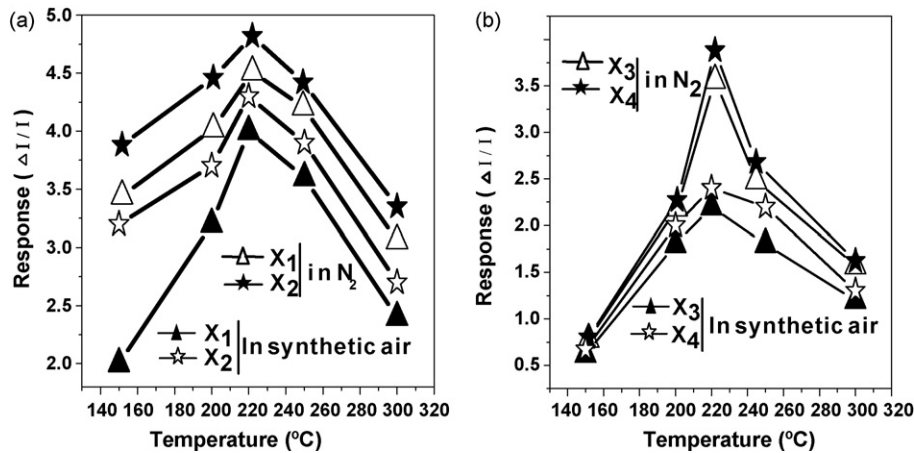


Fig. 6. Response vs. temperature curves of (a) Rh/ZnO/Zn (X_1 and X_2) and (b) Pd-Ag (26%)/ZnO/Zn (X_3 and X_4) MIM sensors in the presence of 1% methane in pure nitrogen and in synthetic air. The response of the sensor is higher in nitrogen than in synthetic air at 0.4 V and at 220 °C for both cases.

10 nm) of the ZnO thin films [19].



When methane is introduced, chemisorbed oxygen reacts with methane and produces CO_2 and H_2O as depicted in reaction (4). The electrons gained by the chemisorbed oxygen become free and come back to the surface of ZnO as a result of which the current through the junction increases.

After the complete removal of the chemisorbed oxygen, methane undergoes dissociative adsorption on the noble metal surface and produces hydrogen. The hydrogen molecules decompose on the catalyst metal surface and the hydrogen atoms are chemically adsorbed on it. The atomic hydrogen diffuses rapidly through the metals due to high solubility and diffusivity of hydrogen through Pd and Rh and are adsorbed on the junction between the noble metal and ZnO where a dipole layer forms and reduces the work function of the metal [20,21]. The lowering of the work function of the metal lowers the barrier height, resulting in an increase in the current through the junction. When methane is introduced into the gas sensing chamber using synthetic air as a carrier gas, the oxygen atoms are chemisorbed on the catalytic metal and an

equilibrium is established between the adsorbate and the adsorbent to a large extent, thereby reducing the adsorption sites for the testing gas and thus yielding less current as compared to methane in a pure nitrogen carrier gas. Further, some of the hydrogen atoms produced by the dissociation of methane molecules react directly with oxygen of synthetic air instead of getting chemisorbed on the catalytic metal to produce H_2O molecules and thus reduce the current through the junction. As a result a lower response to methane is obtained in synthetic air.

As the porous ZnO thin films have a large number of interface states and there is a high electrostatic potential across the junction, the current through the junction in air is very low and the relative change of current in the presence of methane is quite high due to partial passivation of the interface states and lowering of the electrostatic potential, thus giving a high response. The response of the MIM sensors using ZnO thin films grown with UV light is high, mainly due to higher gas adsorption kinetics because of the larger pore diameters.

The transient response of the MIM sensors recorded in the presence of different methane concentrations (0.01%, 0.05%, 0.1%, 0.5% and 1%) at 220 °C and at a 0.4-V bias is shown in Fig. 8. From the figure it is observed that upon exposure to methane the sensor current initially increases and after a while it gets saturated as expected. After cutting off the methane flow the current decreases

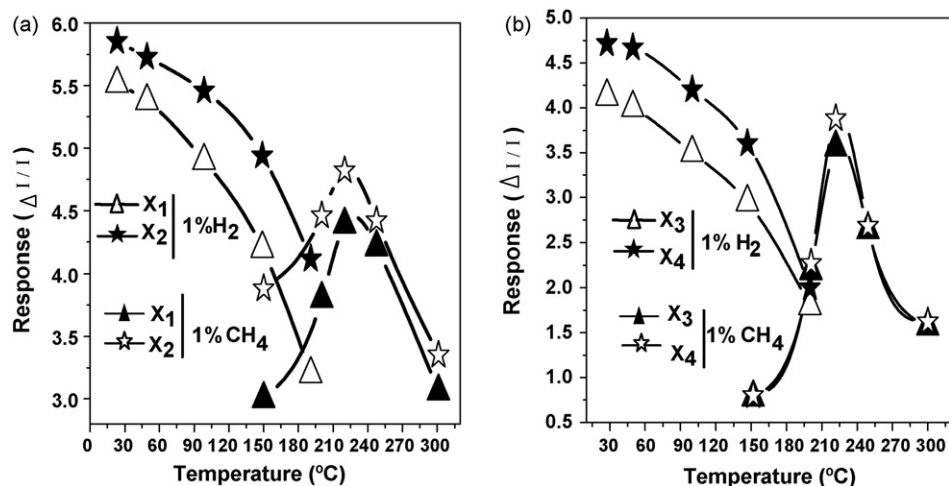


Fig. 7. Response vs. temperature curves of (a) Rh/ZnO/Zn (X_1 and X_2) and (b) Pd-Ag (26%)/ZnO/Zn (X_3 and X_4) MIM sensors in the presence of 1% hydrogen and 1% methane. The maximum response to hydrogen and methane was obtained at 30 °C and 220 °C, respectively, for both cases.

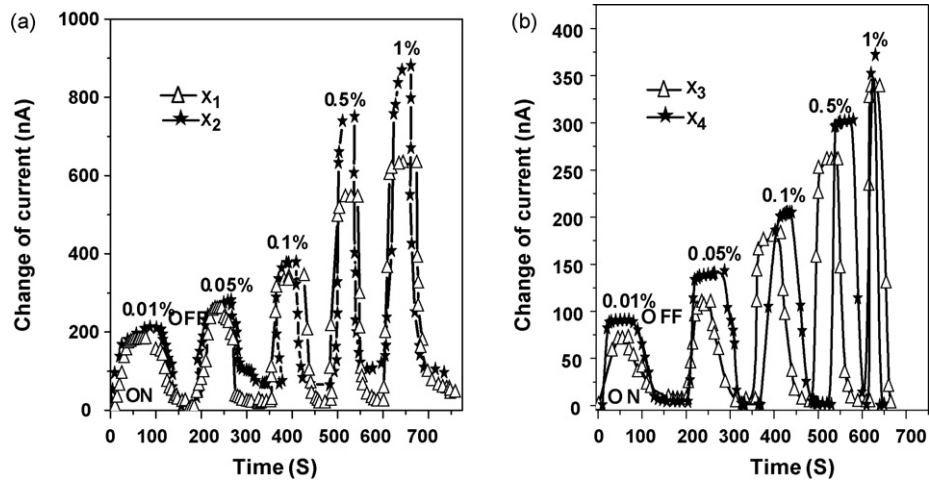


Fig. 8. Transient response curves of (a) Rh/ZnO/Zn (X_1 and X_2) and (b) Pd-Ag (26%)/ZnO/Zn (X_3 and X_4) MIM sensors at 220 °C and at a 0.4-V bias.

Table 2

The results of MIM sensors using ZnO grown without and with UV light

CH ₄ (%)	Without UV						With UV					
	Response		Response time (s)		Recovery time (s)		Response		Response time (s)		Recovery time (s)	
	Rh	Pd-Ag	Rh	Pd-Ag	Rh	Pd-Ag	Rh	Pd-Ag	Rh	Pd-Ag	Rh	Pd-Ag
0.01	1.22	0.99	130.2	22.3	148.3	48.3	1.40	1.00	127.3	19.1	142.6	47.1
0.05	1.82	1.38	52.2	19.7	120.0	46.2	1.89	1.40	47.2	17.2	117.3	46.2
0.1	2.48	2.21	42.8	16.4	89.2	38.1	2.70	2.24	35.0	15.0	80.2	37.1
0.5	3.87	3.11	38.2	12.1	80.2	20.2	4.11	3.18	32.0	8.2	79.2	19.9
1	4.39	3.81	30.2	8.8	78.4	16	4.82	3.85	24.5	5.1	72.3	16.14

and returns almost to the baseline value due to adsorption of oxygen and desorption of hydrogen from the junction producing H₂O in the molecular level. However, some hydrogen molecules remain adsorbed in the junction and cannot desorb instantly and so the current through the junction cannot come back exactly to its original base line value. From the transient response cycles the response and recovery times were calculated and are summarized in Table 2. The response time is defined here as the time for the current to reach 67% of the saturation value. After the gas pulse is cut off the time required for reducing the current to 67% of the saturation value is defined as the recovery time. The response and recovery times of X_2 and X_4 sensors are relatively faster than X_1 and

X_3 sensors. The possible interpretation is that the thickness of the ZnO thin films deposited in the presence of UV light is almost half than the films grown without UV light, resulting in the rapid current flow kinetics. From Table 2 it appears that the response of Rh/ZnO/Zn sensor is high as compared to that of Pd-Ag/ZnO/Zn sensor, but the response and recovery times of the latter are faster. Specially the sensor X_4 shows good response (3.85), faster response time (~5 s) and recovery time (~16 s) as compared to the values reported by others. So the Pd-Ag/ZnO/Zn MIM sensor is suitable for sensing methane of low concentrations at relatively lower operating temperature with appreciably faster response and recovery times.

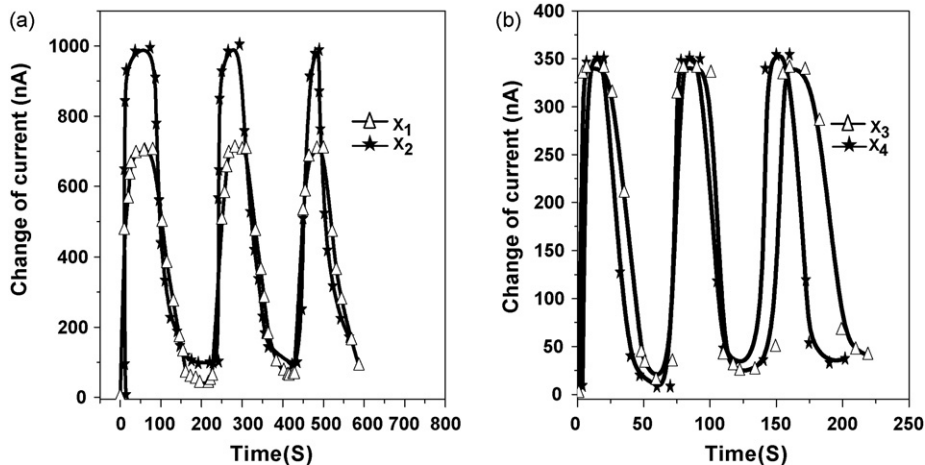


Fig. 9. Repeated cycles of transient response of (a) Rh/ZnO/Zn (X_1 and X_2) and (b) Pd-Ag (26%)/ZnO/Zn (X_3 and X_4) MIM sensors at 220 °C and at a 0.4 V to 1% methane in nitrogen.

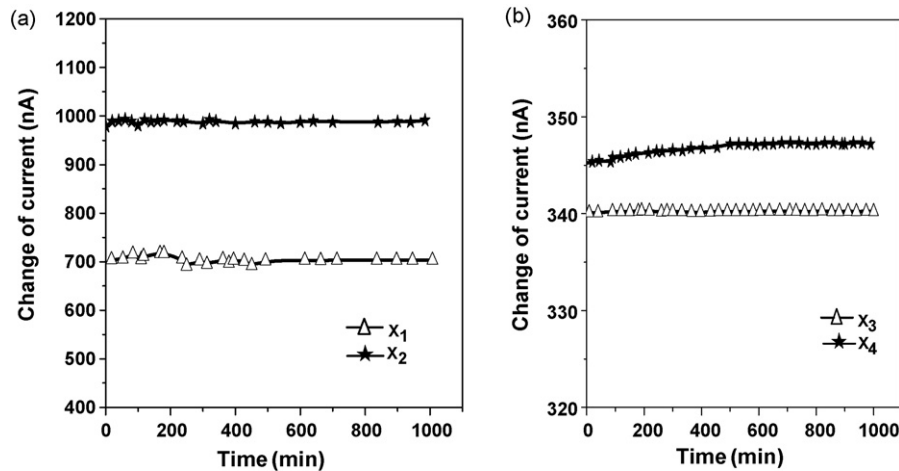


Fig. 10. Stability study of (a) Rh/ZnO/Zn (X_1 and X_2) and (b) Pd–Ag (26%)/ZnO/Zn (X_3 and X_4) MIM sensors at 220 °C at and 0.4V in 1% methane in nitrogen for 16h.

This type of MIM sensors showed reproducible results for the repeated cycles in 1% methane as shown in Fig. 9. It is apparent from the figure that Rh shows appreciable change in the response behavior with ZnO films deposited in the presence of UV light and Pd–Ag shows a little change. We tested the stability of the MIM sensors at 220 °C with a 0.4-V bias and 1% methane, and the results are shown in Fig. 10. It is evident from the figure that after a little initial fluctuation the sensors show more or less stable performance during a period of 16 h with a negligible variation of current.

4. Conclusion

The Rh/ZnO/Zn and Pd–Ag/ZnO/Zn sensors using electrochemically grown ZnO thin films in the absence and presence of UV light were studied with different concentrations of methane. The UV-assisted grown ZnO thin films exhibited high response and fast response and recovery times. Again, compared to the Rh/ZnO/Zn structure, Pd–Ag/ZnO/Zn showed low response and recovery times for sensing 1% or less concentration of methane. The optimum temperature and the optimum voltage for methane sensing were 220 °C and 0.4V, respectively, which are very important for the sensor to operate with low power consumption. Experiments with synthetic air as a carrier gas showed lower performance than in a nitrogen carrier. The selectivity of the sensors for methane sensing in the presence of hydrogen was studied and was found to be more selective with respect to hydrogen only at higher temperature. The stability of both sensor structures was good as was studied for duration of 16 h.

Acknowledgement

The authors thank Mr. S. Maji, IACS, Kolkata, India for FESEM images.

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