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# Low temperature methane sensing by electrochemically grown and surface modified ZnO thin films

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#### ABSTRACT

The functional characteristics of the planar resistive and MIM (metal-insulator-metal) sensors using electrochemically grown nanocrystalline–nanoporous ZnO thin films and surface modified by dipping in an aqueous solution of PdCl<sub>2</sub> were investigated for methane sensing. It was found that the operating temperature was substantially reduced to 70 °C and 100 °C for the two different configurations, respectively, after this interesting and somewhat novel surface modification step. A high purity Zn was anodized to produce ZnO thin films using a Pt cathode, a calomel reference electrode and a 0.3 M oxalic acid electrolyte. Pd–Ag (26%) was used as the catalytic metal contact to ZnO to fabricate a resistive and mIM configuration. The response of the order of ~48, a response time of ~4.5 s and a recovery time of ~22.7 s were obtained for the planar resistive structures, while the MIM structures showed a response of the order of ~32, a response time ~2.7 s and a recovery time of ~16 s. The sensors were studied in the presence of 1% methane in nitrogen and in synthetic air in separate experiments. The performance was somewhat reduced in synthetic air for both the sensor structures while maintaining the optimum operating temperature the same. Both the sensors were stable in 1% methane in nitrogen as well as in 1% methane in synthetic air.

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#### 1. Introduction

Metal oxide nanostructures can work as sensitive and selective chemical sensors. Nanostructural sensor elements can be configured as resistors whose conductance can be modulated by charge transfer across the surface or as a barrier junction device whose properties can be controlled by applying potential across the junction. Functionalizing the surface further offers a possibility to improve their sensing ability.

Continuous research and development activities are being pursued to explore a gas sensor for detection of low concentrations of methane in the coalmine atmosphere at substantially low temperature so that the methane explosion is not further accelerated by the high sensing temperature [1,2]. We have been investigating for the last couple of years on the development of a low temperature methane detector using nanocrystalline ZnO based chemical gas sensors with Pd as the catalytic metal contact on the metal oxide surface. In our previous publications [3–7] we reported the sensing temperature between 210 °C and 250 °C depending upon whether the ZnO sensing film was grown electrochemically or by a sol–gel method, respectively. But these relatively high temperatures for detection of methane are still not suitable for applications in the coalmines. We report in this communication a novel method of reducing methane sensing temperature considerably by the Pd surface modification of the electrochemically deposited nanocrystalline-nanoporous ZnO films using a very low concentration of PdCl<sub>2</sub> solution. We adopted a planar resistive configuration and a kind of metal-insulator-metal (MIM) structure. While the resistive sensor showed the temperature of methane detection at 70 °C the MIM sensor structure recorded the maximum response at 100 °C with a minimum response time of  $\sim$ 2.7 s. Our investigation further demonstrated that the response in synthetic air is also substantially high at the same temperatures for both the structures with a little increase in response time. The stability study for more than 1 month in the presence of 1% methane in nitrogen showed a long term operation of both the sensor structures for more than 30 days without any visible deterioration of the response behaviour.

#### 2. Experimental

The nanocrystalline–nanoporous ZnO thin films were prepared by electrochemical anodization of high purity Zn ( $8 \text{ mm} \times 8 \text{ mm}$ ) (99.9% purity, Aldrich Chemicals, USA) of thickness 0.5 mm using a Pt cathode, a calomel reference electrode, a 0.3 M oxalic acid electrolyte (99%, MERCK, India) and a constant 10 V potentio-





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Fig. 1. XRD of an electrochemically grown ZnO thin film.

static (Scanning Potentiostat-362, Princeton Applied Research, USA) power supply. The separation between two electrodes (cathode and anode) was 18 mm. The calomel reference electrode was placed very near to the anode. The detail preparation and characterizations of the electrochemically grown ZnO thin films were investigated in our earlier publication [8]. The thickness of the ZnO thin films was 8.2 µm as was measured by a surface profilometer (DEKTAK Surface profilers, Sloan Technology, Veeco, Version-1.05). The grown ZnO thin films were dipped into a 0.01 M PdCl<sub>2</sub> solution for 2 s, followed by baking and annealing at 110 °C for 10 min and 300 °C for 30 min, respectively. X-ray diffraction (XRD) (Bruker DH Advance) (Fig. 1), field emission scanning electron microscopy (FESEM) and energy dispersive X-ray (JEOL, JSM-6700F) (Fig. 2) were employed to study the polycrystalline nature, crystal size, surface morphology and pore size of the deposited ZnO thin films. FESEM pictures also demonstrate the nanocrystalline and nanoporous characters of the ZnO surface, both unmodified and modified with dispersed Pd nanoparticles. The schematics of the planar and MIM structures are shown in Fig. 3. The Pd-Ag (26%) catalytic metal contact of thickness 0.2 µm was deposited on the Pd modified ZnO thin films by e-beam evaporation using an Al metal mask and the electrical contacts were taken by using fine



Fig. 2. (a and b) FESEM images and (c and d) EDX spectra of unmodified and Pd-modified ZnO thin films, respectively.



Fig. 3. The schematics of (a) the planar and (b) the MIM sensor structures.

copper wire and silver paste. The gas sensor characteristics in the absence and presence of different concentrations 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  $\pm 1$  °C by a precise temperature controller with a built-in copper constantant thermocouple. For sensor study high purity methane, high purity nitrogen and synthetic air were used. To measure and control the flow rates of the gases precisely throughout the experiments, mass flow meters and mass flow controllers (Digiflow, USA) were used. A Keithley picoammeter with a voltage source (Model 6487) was used to study the current–voltage characteristics of the sensor structures in the absence and presence of methane.

#### 3. Results and discussions

From Fig. 1 it is observed that the grown ZnO thin film is polycrystalline in nature with preferential growth along (101) and (110) directions. The average crystal size, calculated from the Scherer formula, was 2.4 nm. From FESEM the surface morphology and the pore size were studied for both unmodified and modified ZnO surfaces, and it was observed that the pore size varied from 19 nm to 35 nm for the unmodified ZnO surface and 10 nm to 20 nm for the modified one after treatment with the PdCl<sub>2</sub> solution (Fig. 2a and b). It can be observed that there was little difference between FESEM pictures of unmodified and Pd-modified ZnO films, but EDX (Fig. 2c and d) figures indicate clearly the presence of Pd in the modified film.

The sensors were characterized with different concentrations of methane gas in pure nitrogen and also in synthetic air in the temperature range 50-150 °C. The current–voltage characteristics of both types of sensor structures were studied at different temperatures and the magnitude of response (*S*) was calculated using the relation,

$$S = \left\{ \frac{I_{\rm g} - I_{\rm a}}{I_{\rm a}} \right\}_V \tag{1}$$

where  $I_g$  = the current in the presence of gas and  $I_a$  = the current in air.

The change of response with temperature and with the bias voltage are shown in Fig. 4a and b, respectively, and it can be observed



Fig. 4. (a) Response vs. temperature curves at 3 V and (b) response vs. voltage curves for both types of sensor structures.



Fig. 5. Response vs. temperature curves of (a) the resistive planar and (b) the MIM sensor structures at 3 V in the presence of 1% methane in pure nitrogen and in synthetic air.



Fig. 6. Response vs. temperature plots of (a) the resistive planar and (b) the MIM sensors in 1% H<sub>2</sub> and 1% CH<sub>4</sub> in pure N<sub>2</sub> environment.

from Fig. 4a that the maximum response is obtained at 70  $^{\circ}$ C and 100  $^{\circ}$ C for the planar and the MIM sensor structures, respectively. It is observed from Fig. 4b that for both types of structures the maximum response occurs at 3 V bias. Fig. 5a and b shows the response vs. temperature curves for both planar and MIM structures, respectively, with 1% methane in pure nitrogen and in synthetic air, and

it is found that the response is higher in pure nitrogen than in synthetic air for both types of sensor structures. Fig. 6 shows the response vs. temperature curves of both types of sensors at different operating temperatures using 1% hydrogen and 1% methane in pure N<sub>2</sub> environment. Hydrogen shows the maximum response at 50 °C for both planar and MIM sensors. The selectivity of methane



Fig. 7. Transient response curves of the resistive planar structure in 1% methane using (a) nitrogen and (b) synthetic air as a carrier gas.



Fig. 8. Transient response curves of the MIM structure in 1% methane using (a) nitrogen and (b) synthetic air as a carrier gases.

![](_page_4_Figure_3.jpeg)

Fig. 9. Concentrations vs. response curves of both the resistive planar and the MIM structures using (a) nitrogen and (b) synthetic air as a carrier gas.

![](_page_4_Figure_5.jpeg)

Fig. 10. Methane concentrations vs. time curves of both the resistive planar and the MIM structures using (a) nitrogen and (b) synthetic air as a carrier gases.

Table 1

The results of the planar and MIM sensor structures in different concentrations of methane using nitrogen as a carrier gas at  $70 \,^{\circ}$ C and  $100 \,^{\circ}$ C, respectively

% of methane	Response		Response time (s)			Recovery time (s)	
	Planar	MIM	Planar	MIM	_	Planar	MIM
0.01	21.2	18.2	16.2	10.2		33.2	18.7
0.05	25.4	22.3	13.3	6.7		30.3	17.2
0.1	39.3	23.2	11.1	6.03		27.5	16.8
0.5	42.2	25.2	8.4	4.02		25.7	16.1
1	47.5	32.2	4.6	2.69		22.7	16.0

in the presence of hydrogen is improved at the optimum operating temperatures of 70 °C and 100 °C for planar and MIM sensors, respectively. However, the MIM sensor shows much better selectivity for methane. Fig. 7a and b represent the transient responses to different concentrations of methane at 70 °C of the resistive planar structure using nitrogen and synthetic air as a carrier gas, respectively. Similarly the response behaviour of the MIM structure for methane at 100 °C is shown in Fig. 8 for both types of carrier gases. The response as a function of methane concentration for both the planar structure operating at 70 °C and the MIM structures operating at 100 °C with two different carrier gases of nitrogen and synthetic air are displayed in Fig. 9. The response and recovery times with increasing methane concentrations for both the sensor structures as derived from the transient response curves (Figs. 7 and 8) are shown in Fig. 10.

Experiments on repeated cycles of the transient responses for both planar and MIM sensor structures are presented in Fig. 11 in order to show the reproducible behaviour of the sensors. Fig. 12 presents the stability of both types of sensor structures. While for 1% methane with a nitrogen carrier gas the study was continued for 132 h with a duration of 4 h per day in 33 days, the same experiment with synthetic air as a carrier gas was conducted for 8 h with a duration of 2 h per day in 4 days. The planar and MIM sensors were operated at 70 °C and 100 °C, respectively, and at 3 V. Finally Tables 1 and 2 summarize the results for sensing 1% methane in nitrogen and in synthetic air, respectively.

The Pd loaded nanocrystalline (below 10 nm) ZnO enhances the oxygen spillover process [9,10], resulting in a large amount of chemisorbed oxygen which yields a high electrostatic potential across the Pd–Ag/ZnO Schottky interface [6,7]. Subsequently methane reacts with this adsorbed oxygen and produces  $H_2O$  and  $CO_2$ , as shown in Eq. (2) below

$$CH_4 + 2O_2^- \leftrightarrow CO_2 + 2H_2O + 4e^-$$
(2)

The electrons on the surface of ZnO enhance the current through the electrodes. The detailed mechanism of the gas sensing of Pd–Ag/ZnO Schottky contacts using electrochemically grown ZnO thin films was reported in our earlier publications [6,7]. Since the MIM sensor has only one Schottky contact (Pd–Ag/ZnO), the current through the electrodes is controlled by the Pd–Ag/ZnO junction. On the other hand, the planar structure which has two-Schottky contacts with double barrier junctions and a large exposed surface area

#### Table 2

The results of the planar and MIM sensor structures in different concentrations of methane using synthetic air as a carrier gas at 70 °C and 100 °C, respectively

% of methane	Response		Response time (s)			Recovery time (s)	
	Planar	MIM	Planar	MIM	_	Planar	MIM
0.01	9.2	6.5	19.2	13.2		34.2	20.3
0.05	14.6	11.4	17.4	9.3		32.8	19.2
0.1	18.3	16.8	16.4	8.2		29.6	17.3
0.5	21.6	17.2	15.2	6.9		27.4	17.1
1	25.7	20.4	12.6	4.2		23.4	16.2

where oxygen atoms are directly chemisorbed, gives a lower current than the MIM sensor in air. Therefore the change of current in the presence of methane is relatively higher (Fig. 4) for the planar structure than the MIM one. It is further observed from Fig. 4 that the temperature for maximum response was reduced to 70 °C for the planar and 100 °C for the MIM structure. The possible reason for the response at substantially low temperature may be mentioned as due to the increased surface free energy of the nanocrystalline ZnO surface [11]. It is known that with an increase in gas adsorption, the catalytic metal electrode (Pd) work function decreases, and the current through the Pd-ZnO Schottky junction increases, and thus the gas response increases [6,7]. Once the desorption starts at higher temperature, the change in work function decreases and the current in the presence of gas  $I_{g}$  also decreases, and so also the response. Since the thickness of the ZnO film (8 µm) is quite large, the electrons have to migrate through the large insulating layer in order to conduct via the bottom metal substrate. In the planar configuration electrons would prefer to conduct from one electrode to other through the Pd modified ZnO surface, where Pd can assist relatively faster conductivity through possible hopping conduction mechanism from one island to other of discontinuously dispersed Pd. For nanocrystalline structure the adsorption activation energy is considerably low and the presence of dispersed Pd nanoparticles over the ZnO surface further reduces the adsorption energy. As a result the sensors respond at considerably low temperature. The favourable adsorption kinetics due to reduced adsorption activation energy also exhibits a remarkably short time of response. The longer recovery time is presumably due to the lower desorption rate at low temperature. It is worth mentioning here that imperfect structural orientation of the polycrystalline sensing layer (ZnO) also modulates the gas adsorption behaviour and thus the sensing parameters.

In the case of synthetic air as a carrier gas, oxygen is parallely chemisorbed on the Pd catalysed ZnO surface and there is a competitive equilibrium between oxygen and methane as the adsorbates on ZnO. As a result it may well be depicted that the adsorption sites for methane is reduced and chemisorbed oxygen reacts with hydrogen instead of getting diffused into the catalytic metal to produce H<sub>2</sub>O molecules and thus reduce the current through the electrodes. Therefore a lower response to methane is obtained in synthetic air. The transient responses of the planar and MIM sensors recorded in the presence of different methane concentrations (0.01%, 0.05%, 0.1%, 0.5% and 1%) at 70 °C and 100 °C and at 3V bias are shown in Figs. 7 and 8, respectively. From the figures it is observed that initially the current increases in the presence of methane and then gets saturated. After withdrawing the methane flow the current decreases and returns almost to the baseline value. From the adsorption-desorption kinetics it can be explained that desorption always takes place at higher temperature than adsorption. So, some of hydrogen atoms obtained from methane molecules through dissociative chemisorption gets permanently adsorbed on nanoporous ZnO, and as a result the current through the electrodes cannot come back exactly to its original base line value. The response, response time and recovery time were calculated and are shown in Figs. 9 and 10, and the results are also summarized in Tables 1 and 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. From Fig. 9a it can be observed that the response increases up to 1000 ppm of methane and then it slowly gets saturated most probably due to the fact that the active surface area is already occupied and so a further increase of methane concentration does not change the response of the sensors. This is further supported by Christmann et al. [12] who clearly indicated that with increasing coverage ( $\theta$ ) the

![](_page_6_Figure_1.jpeg)

Fig. 11. Repeated cycles of the transient response for (a) the planar and (b) the MIM sensor structures for reproducibility study.

sticking coefficient went down initially sharply and finally tended towards saturation. From Fig. 9b and Tables 1 and 2 it is clear that the response and recovery times of the MIM sensor are relatively shorter than the planar sensor. The possible interpretation is that the separation between two electrodes for the planar configuration is larger (2 mm) than the MIM (8  $\mu$ m) configuration, resulting in the rapid current flow kinetics.

There are very few reports on metal oxide based methane gas sensors with high response and shorter response and recovery times. So far the reported sensor structures based on oxide semiconductors operating in the resistive mode and at high temperature showed relatively longer response (>10s) and recovery times (>40s). In this investigation, particularly the MIM sensor showed good response (~32), shorter response (~2.69s) and recovery times (~16s) as compared to the values reported by others [1–7,13–18]. So, the Pd–Ag/ZnO/Zn MIM sensor with a ZnO film deposited electrochemically and surface modified with Pd is suitable for sensing low concentrations of methane at relatively lower operating temperature (100 °C) with appreciably shorter response and recovery times. It is also apparent from Tables 1 and 2 that the response time is shorter than the recovery time. When methane supply is cut off and air is allowed to flow into the sensing cham-

ber, oxygen is adsorbed on crystalline ZnO by spillover technique as mentioned above and the out diffusion of adsorbed hydrogen from the junction takes place simultaneously. The chemisorbed oxygen reacts with hydrogen and produces  $H_2O$ . As a result electrons of the chemisorbed oxygen come back to the surface of ZnO and the current through the electrodes increases instead of decreasing. This process continues until all the atomic hydrogen desorbs from the junction and therefore the recovery time is longer than the response time. This was corroborated by Christmann et al. [12].

Both the sensors show temperature dependent selectivity for methane and hydrogen in the presence of each gas. For the planar resistive sensor the hydrogen response is maximum at  $50 \,^{\circ}$ C with a very low response for methane. At  $70 \,^{\circ}$ C both hydrogen and methane respond almost to the same extent. On the other hand, the MIM sensor shows distinct selectivity for methane at  $100 \,^{\circ}$ C where hydrogen response is almost zero.

The sensors showed reproducible results for the repeated cycles in 1% methane, as shown in Fig. 11. We tested the long-term stability for 33 days for the planar and MIM sensors at 70 °C and 100 °C, respectively, with a 3 V bias and 1% methane, and the results are shown in Fig. 12. It is evident from the figure that after a little initial fluctuation, the sensors show more or less stable performance with

![](_page_6_Figure_8.jpeg)

Fig. 12. Stability study of both types of sensor structures (a) using nitrogen and (b) synthetic air as a carrier gas.

a negligible variation of current. We also tested the stability of both the sensor structures with synthetic air as a carrier gas for 8 h, as shown in Fig. 12b and we found a good stability.

#### 4. Conclusion

Our investigations on methane sensing using nanocrystalline-nanoporous ZnO thin films, deposited by an electrochemical method and surface modified by Pd nanoparticles using chemical dipping procedure, reduced the temperature and response time of methane detection to an appreciably low value for both a planar resistive and an MIM sensor. For nanocrystalline structure the adsorption activation energy was considerably low and the presence of dispersed Pd nanoparticles acting as a catalyst over the ZnO surface further reduced the adsorption energy. As a result the sensors responded at considerably low temperature. However, the MIM structure operating at 100 °C showed the minimum time of response and a good selectivity for methane in the presence of hydrogen. The stability of both types of sensors, in the presence of 1% methane in nitrogen as a carrier gas, was studied for more than 30 days and it was found to be excellent. The stability in 1% methane in synthetic air as tested for 8h was also good.

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