Micromached MoO$_3$ Gas Sensor with Low Power Consumption of 0.5 Watt

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(Received May 3 2005, Accepted August 10 2005)

A new MoO$_3$ based microsensor with low power consumption was presented. Typical size of sensor was 5 mm in width and 8 mm in length. As a sensitive electrode, MoO$_3$ was successfully fabricated by IC technology on pyrex glass of 250 μm in thickness. After annealing at 550 °C for 3 hrs, the film was fully crystallized and demonstrated as pure MoO$_3$ structure. The grain size of MoO$_3$ was plat like and typical size was about 1μm. Based on the results of sensitivity measurement, MoO$_3$ microsensor shows especially high selectivity to H$_2$ reducing gas atmosphere. The applied heater power was lower than 0.5 Watt.

Keywords : MoO$_3$ gas sensor, MEMS, IC technology

1. INTRODUCTION

Current development in gas sensor research strive towards low power consumption, low operating temperature, better selectivity, sensitivity, long terms stability and smaller size. The search for smart sensors has stimulated microelectronics evolution with the development of new electrode materials, sensor structure, and IC technology. Numerous efforts have so far been directed to improve sensitivity and selectivity of semiconductor gas sensors. Loading of a novel metal and/or an oxide on the sensor materials is one of the most important techniques for improving the reactivity. MoO$_3$ has been well known from applications in the field of catalysis for oxidation reactions of hydrocarbons and ethanol[1] and electromagnetism[2]. Its high reactivity is based on the fact that it can be easily reduced. Recently semiconducting MoO$_3$ has been developed as a new gas sensing element[3,4] because of its high reactivity and easy reduction. The MoO$_3$ is an n-type semiconductor with an oxygen deficiency. The band gap is 3.2 eV and the electrical resistivity at room temperature is the order of $10^{10}$ Ω·cm, measured on a sintered pellet[5].

In this paper, micromachined MoO$_3$ sensor with low power consumption and smaller size was fabricated by IC technology in order to meet current sensor development. Thin film consisting of MoO$_3$ nanoparticle was deposited on pyrex glass of 250 μm thickness by DC sputtering technique. It is very important to apply the heater power directly to electrode and temperature detector by using very thin membrane of 250 μm thickness. The sensor consists of three main blocks: interdigitated electrode, heater and temperature detector. Typical sensor size was 5.0 mm in length and 3.8 mm in width. Fabricated microsensor was systematically evaluated in terms of different reducing gas such as H$_2$, ethanol, methanol and acetone with different gas concentration. Also gas sensitivity was systematically monitored as a function of heater power consumption.

2. EXPERIMENTAL

The sensor was fabricated by using conventional silicon IC technology. The microsensors were fabricated by photolithography with 4 metal masks and a shadow mask. Initially 1000 Å Pt was deposited by DC sputtering on the back of pyrex glass substrate for heater and temperature detector. After lift-off of heater and temperature detector interdigitated Mo films for
Table 1. Deposition conditions.

<table>
<thead>
<tr>
<th>Deposition Condition</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>- basal pressure (torr)</td>
<td>$2 \times 10^{-6}$</td>
</tr>
<tr>
<td>- working pressure (morr)</td>
<td>2</td>
</tr>
<tr>
<td>- distance between target and substrate</td>
<td>20 cm</td>
</tr>
<tr>
<td>- deposition temperature ($^\circ$C)</td>
<td>room</td>
</tr>
<tr>
<td>- working gas</td>
<td>100% Ar</td>
</tr>
<tr>
<td>- substrate</td>
<td>pyrex glass (250 $\mu$m)</td>
</tr>
<tr>
<td>- film deposition time</td>
<td>60 min</td>
</tr>
<tr>
<td>- film thickness (Mo)</td>
<td>2000 Å</td>
</tr>
</tbody>
</table>

Fig. 1. Diagram of sensor design.

electrode was made by means of rf magnetron sputtering. The deposition was performed under 100% Argon atmosphere for 60 min on pyrex glass substrate of 250 $\mu$m thickness. The working and basal pressure was maintained at a constant level of 2 mtorr and 2 $\times 10^{-3}$ mtorr respectively. The film thickness was around 2,000 Å. The film deposition condition was summarized in Table 1.

After sputtering the Mo film was annealed in air at 550 $^\circ$C for 3 hrs. Typical size and structure of microsensor are shown in Fig. 1 and 2. Figure 1 and 2 shows the schematic diagram of sensor design and the typical sensor structure. The cross sectional overview, interdigitated electrode, heater and temperature detector are presented in Fig. 1(a), (b) and (c) respectively. The gases sensing experiments were carried out in a computer-controlled gas measurement system. The measurement atmosphere is provided by a gas mixing system, based on mass flow controllers. The gas under test were H$_2$, acetone, methanol and ethanol at operation temperature between 100 and 250 $^\circ$C. Normal applied power for heater was about 0.45 Watt.

![Image](image1.png)

Fig. 2. The typical sensor structure; (a) cross sectional overview of sensor, (b) interdigitated electrode and MoO$_3$ film, and (c) heater and temp. defector.

3. RESULTS AND DISCUSSION

After annealing at 550 $^\circ$C for 3 hrs, the film was fully crystallized and demonstrate pure MoO$_3$ structure without indicating any second phases. Figure 4 shows SEM surface morphology of MoO$_3$ film after annealing.

The film morphology was uniformly dense and plate-like structure. The typical grain size was about 1 $\mu$m. The gas sensing properties was evaluated in terms of different gas atmospheres. Figure 5 demonstrates the gas sensing behavior to H$_2$ gas in terms of different concentration.

The response time of a sensor to H$_2$ gas is directly related to gas detection and classification. The MoO$_3$ microsensor shows high sensitivity and selectivity to H$_2$ gas. Initial response time to H$_2$ gas of 100 ppm was
Fig. 3. XRD patterns of MoO₃ film annealed at 550 °C for 3 hrs.

Fig. 4. SEM image of MoO₃ film.

Fig. 5. Gas sensing characteristics in terms of different H₂ concentration.

180 sec. However the sensor response time increased rapidly with increasing the gas concentration from 100 ppm to 500 ppm. Typical the response time to H₂ gas with 500 ppm was about 30 sec after starting the gas detection. The output voltage signal indicating sensor selectivity is very high.

Fig. 6. Gas sensing characteristics in terms of different heater power.

Fig. 7. Gas sensing characteristics in terms of acetone and ethanol.

Fig. 8 Gas sensing characteristics in terms of acetone, ethanol and methanol concentration.
Figure 6 shows the sensitivity dependance in terms of applied heater power. The gas concentration of \( \text{H}_2 \) was kept at 1000 ppm. As can be seen in Fig. 6, the sensitivity relatively increased with increasing heater power from 0.3 to 0.45 Watt. Normally in most cases, desorption and adsorption behavior become faster with increasing the applied power. In Fig. 6, the desorption behavior at 0.45 Watt is much faster than absorption at 0.3 Watt and thereby sensitivity decreased.

Figure 7 demonstrates the gas sensing characteristic when sensor was exposed to acetone and ethanol with 500 ppm. In Fig. 7, the response time to acetone of 500 ppm concentration was about 15 sec while the response time to ethanol of 500 ppm concentration was about 18 sec. As compared with the sensing behavior to \( \text{H}_2 \) gas in terms of the output signal, the sensitivity is high but the recovery time was somewhat low.

Figure 8 represents gas sensing characteristics in terms of acetone, ethanol and methanol. Typical response time to methanol with 500 ppm was 20 sec whereas the normal response time of ethanol was about 18 sec.

The sensing behaviors exposed to 3 different gases such as acetone, ethanol and methanol are quite similar to each other except indicating the low selectivity in methanol atmosphere.

Based on the results of sensing measurement, the \( \text{MoO}_3 \) microsensor showed the excellent selectivity to \( \text{H}_2 \).

4. CONCLUSION

Thin film sensor of \( \text{MoO}_3 \) was successfully fabricated by IC technology on pyrex glass of 250 \( \mu \text{m} \) in thickness. The film morphology was uniformly dense and showed the plate-like structure and typical grain size was about 1 \( \mu \text{m} \). Typical size of sensor was 5 mm in width and 8 mm in length. \( \text{MoO}_3 \) film gas sensor showed especially high selectivity to \( \text{H}_2 \), reducing gas atmosphere. The applied heater power was lower than 0.5 Watt. Typical response time to \( \text{H}_2 \) gas with 500 ppm was about 30 sec after starting the gas detection. Also it was found that the sensing behaviors to acetone, ethanol are quite similar to each other while the selectivity in terms of methanol atmosphere is somewhat low.

ACKNOWLEDGMENTS

This work was supported by Korea Research Foundation(KRF 2003-002-D00160).

REFERENCES