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Fabrication and Characterization of MEMS Based Optical Hydrogen Sensors

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Abstract

This paper presents the fabrication and characterization of MEMS based optical type hydrogen sensors using transparent 3C-SiC membrane and photovoltaic effect. 3C-SiC membrane was fabricated by anisotropic etching. Gasochromic material of Pd was deposited by sputter on the 3C-SiC membrane for gas sensing layer. Pd and WO_3 changes to transparency by exposure to hydrogen and the variations of light intensity generate the photovoltaic of P-N junction between N-type 3C-SiC and P-type Si. Photovoltaic increased with increase of hydrogen concentration. Pd/WO_3 shows almost 2 times faster response and recovery toward hydrogen compared with Pd. However, low response factor is shown at Pd/WO_3 by low penetration ratio.

Key words: Optical type hydrogen sensor, 3C-SiC thin film, Photovoltaic, Pd, WO_3

Introduction

Due to the energy crisis related to the limited resources of fossil fuels, hydrogen is considered the most attractive energy resources in the future for its high efficiency, renewability, and the fact that it is environmentally friendly [1]. Hydrogen is currently used as a fuel for internal-combustion engines and fuel cells, which may soon become ubiquitous in automobiles and homes due to the potential for substantially cleaner emissions when compared to industrial combustion engines [2]. However, general hydrogen sensors such as catalytic, electrochemical and semiconductor sensors use electrical leads, which may induce sparks and provide a cause for ignition in hazardous atmospheres. Therefore, optical type hydrogen sensors based on fiber optics allow working in an explosive environment thanks to the possibility of separating the sensing point from the electrical readout [3]. Various strategies have been proposed, which basically infer the hydrogen concentration from a change in the optical response e.g. the interference pattern, the frequency of the optical signal, or in the intensity of the optical signal. However, optical signal measurement system is expensive and huge. So, portability is restricted. Moreover, low mechanical strength of optical fiber is limiting the portable sensor application. 3C-SiC is interesting material for two third generation photovoltaic (PV) material [4]. Recently, photocapacitance properties of 3C-SiC/p-Si structure were studied [5]. These properties can be utilized as light detecting parts. Changes of voltage can be achieved from variations of light intensity according to hydrogen concentration. Also, chemical stability and transparent of 3C-SiC membrane can be replace the optical fiber which exposure to humidity and hydrogen make color change and crack by decomposition (SiO_2+H_2O->Si-O-HH-O-Si).

In this work, chemically stable 3C-SiC membrane and 3C-SiC/P-Si structures were used optical type hydrogen sensor as sensing part and detection part, respectively. After integrate the light source with sensing part, hydrogen sensing characteristics are evaluated with the hydrogen concentration of 2~10%.

Experimental

Two kinds of 3C-SiC on substrates are employed for fabrication of sensing and detection parts, respectively. 2 µm 3C-SiC thin film deposited on the oxide wafer was used for sensing part. 3C-SiC membrane fabricated by photolithography and back side anisotropic etching in TMAH $(CH_3)_4NOH$ for 16 hours. Gasochromic materials of Pd and Pd/WO_3 deposited on 3C-SiC membrane by sputtering. Pd and WO_3 were deposited 20 sec with 100 W and 60 sec with 50 W, respectively. After deposition of catalysts, 3C-SiC membrane integrated with LED light source. 0.3 µm 3C-SiC thin films on P-type Si were used as detection

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part. For measure of photovoltaic effect at 3C-SiC and P-type Si, Al electrodes were deposited the surface of 3C-SiC and back side of Si. 100% hydrogen mixed with N_2. Net hydrogen concentrations range was from 2~10%. Hydrogen sensing properties are analyzed in dark chamber. Fig. 1 shows the schematic diagram of light source integrated with sensing part and measurement system.

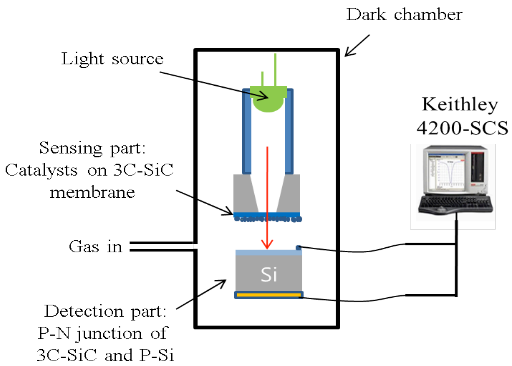

Fig. 1. Schematic diagram of MEMS based optical type hydrogen sensors and their measurement system.

Results and discussion

One cycle response with catalysts and hydrogen concentration are shown in Fig. 2 (a) Pd and (b) Pd/WO_3, respectively.

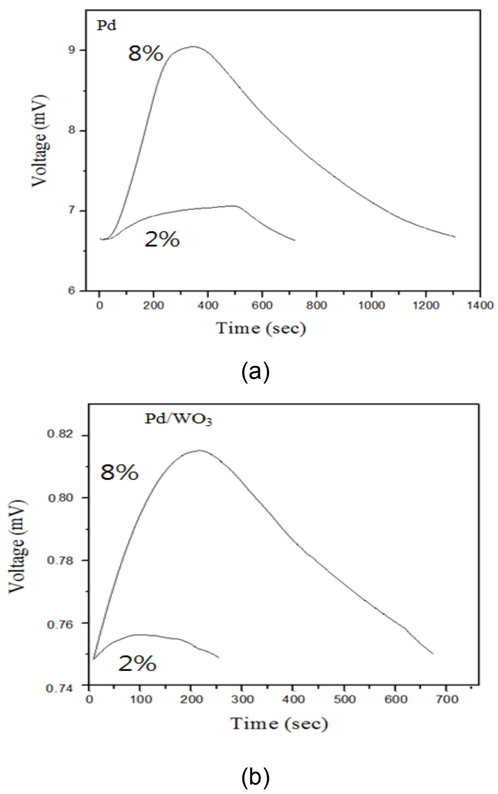

Fig. 2. Responses of the sensors toward different H_2 concentrations at room temperature: (a) Pd and (b) Pt/WO_3.

Gas chromic materials of Pd and WO_3 are changed to transparency when exposure to hydrogen. Thus, generated voltage from P-N junction of 3C-SiC and Si increased with hydrogen on and decreased hydrogen off. Pd/WO_3 shows low photovoltaic compared with Pd only. These come from two layer of Pd/WO_3 makes low penetration ratio. There is almost no baseline shift in the sensor signal.

Fig. 3 shows the photovoltaic variations of optical hydrogen sensors coated with (a) Pd and (b) Pd/WO_3 according to hydrogen concentration. In case of Pd, over than 6% hydrogen concentration, variation of output voltage is decreased compared with 2~4%. However, Pd/WO_3 shows linear response with hydrogen concentrations. These come from phase transition. Phase transition from α to β is shown after 6% in Fig. 3 (a) [6]. However, Pd catalyst increased reaction of WO_3 with hydrogen in Fig. 3 (b) [7].

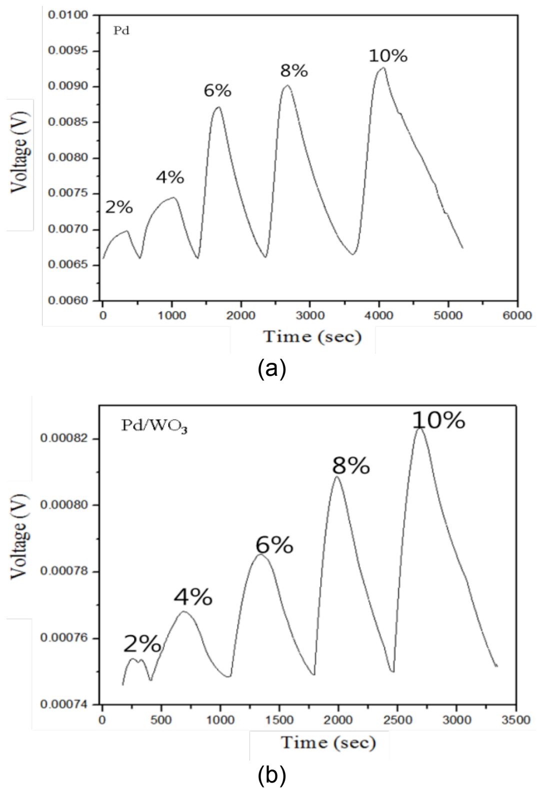

Fig. 3. Photovoltaic variations of optical hydrogen sensors coated with (a) Pd and (b) Pd/WO_3 according to hydrogen concentration.

Fig. 4 (a) and (b) shows response time (time to reach 90% of the maximum transition value) and the recovery time (time to reach 90% of the initial value) of Pd and Pd/WO_3, respectively. Response and recovery time increased with increase of hydrogen concentrations. At 10% hydrogen concentration, response and recovery time of Pd was 300, 900 sec and Pd/WO_3 was 180, 500 sec, respectively.

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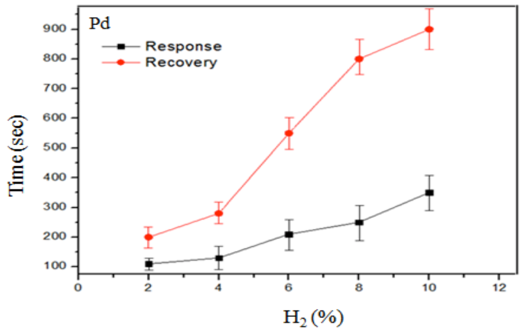
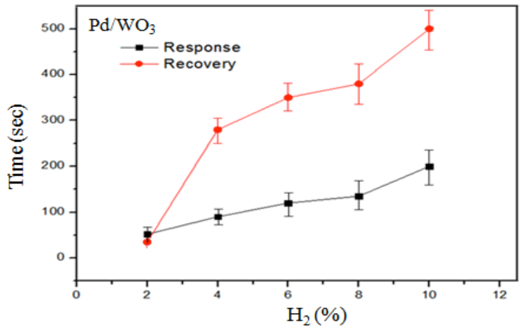


(a)



(b)

Fig. 4. Response and recovery time of Pd and Pd/WO3 with hydrogen concentration.

Tab. 1 shows response factors of Pd and Pd/WO_3, respectively. The response factor can be considered as an indirect measurement of the device sensor signal. The response factor was calculated as follows:

$$\text{Response factor (\%)} = \left(\frac{V_{initial} - V_{response}}{V_{initial}}\right) \cdot 100$$

where $V_{initial}$ is the voltage measured in air and $V_{response}$ is the voltage at each hydrogen concentrations. Response factor increased with the increase of hydrogen concentrations. However, Pd/WO_3 structure shows low response factors by low penetration ratio. If thickness of Pd/WO_3 will optimize, out signal of photovoltaic should be improve.

Conclusions

In this work, optical type hydrogen sensors using 3C-SiC membrane and photovoltaic were fabricated and evaluated. 3C-SiC membrane was integrated with LED light source and P-N junction of SiC/Si was used as a detection parts. Pd and Pd/WO_3 were used as sensing materials and detected hydrogen range from 2~10%. In case of Pd, output signal decreased at high concentration of hydrogen by phase transition. Pd/WO_3 shows linear response signal with hydrogen concentrations. However, Pd/WO_3 structure shows low response factors compared with Pd. This means the Pd/WO_3 has more low penetration ratio compared with Pd. If thickness of Pd/WO_3 will optimize, out signal of photovoltaic should be improve. Novel optical type hydrogen sensor based on 3C-SiC membrane and photovoltaic effect can be applied to high concentration of hydrogen and VOC sensors.

Tab. 1: Variation of response factor (%) with hydrogen concentration.

H_2 (%)	Pd	Pd/WO_3
2	7.69	1.33
4	12.30	4
6	34.61	5.33
8	38.46	7.33
10	42.3	10

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