Sensors and Actuators B 219 (2015) 10–16

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

journal homepage: www.elsevier.com/locate/snb

Electrostatic gas sensor with a porous silicon diaphragm

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ARTICLE INFO

Article history:
Received 21 October 2014
Received in revised form 16 April 2015
Accepted 28 April 2015
Available online 19 May 2015

Keywords:
Porous silicon
Sensors
Diaphragm
Resonance frequency
Frequency response
Electrostatic gas sensor

ABSTRACT

An electrostatic gas sensor with a porous silicon diaphragm was fabricated, and its sensing characteristics towards isopropyl alcohol (IPA) as a model gas were examined. The DC-biased diaphragm in the sensor vibrated under an alternating electric field in a frequency range of 0.5–40 kHz. For the annealed porous silicon diaphragm, four strong resonance peaks were observed in the frequency response. Furthermore, the frequency response properties, which changed with exposure to IPA gas, were used as response signals of the electrostatic gas sensor. As a function of IPA gas concentration, the vibration amplitude increased non-linearly, but the resonance frequency was shifted linearly. Furthermore, the operating voltage dependencies on the vibration amplitude of the diaphragm were investigated, and directions for future study to enhance sensor performance were also discussed.

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1. Introduction

Porous silicon is prepared by electrochemical etching of a single-crystal silicon wafer using an electrolyte containing hydrofluoric acid (HF). This material has a high specific surface area of approximately 600 m^2/cm^3 due to its nanometer-sized pores and high porosity [1,2]. Various applications such as gas sensors [3–5], biosensors [6,7], and drug deliveries [8,9] have been explored by many research groups in efforts to exploit the unique properties of this material. In particular, porous silicon sensors have been widely studied in recent years. Interest in these sensor stems from the excellence of the physical or chemical absorption of a target gas onto the surface of porous silicon due to the high surface area and surface activity. Furthermore, it is well known that capillary condensation, which can enhance sensing capability, occurs in the nanometer-sized pores [10,11].

Various physical properties that are changed by chemical gases have been used as response signals of porous silicon sensors. First, it is well known that the intensity of photoluminescence (PL) emitted from porous silicon by excitation light varies after exposure to organic vapors [12,13]. Moreover, the surface reflectivity of the porous silicon layer is changed by chemical gases [5,14]. This property is widely exploited because measurement of reflectivity change is easier than that of PL intensity change. Finally, the electrical conductivity change of the porous silicon layer by chemical gases is also used as a response signal [15,16]. The sensor based on this phenomenon is advantageous in terms of signal processing since the output of the sensor is an electrical signal. However, the difficulty of making electrical contact between the electrodes and the fragile porous silicon surface is a notable disadvantage.

In this paper, we propose a novel electrostatic gas sensor that is different from conventional porous silicon-based sensors that operative based on changes of PL intensity, reflectivity, or conductivity, as described above. The isopropyl alcohol (IPA) gas sensing performance of the fabricated sensor system was evaluated. We chose IPA gas as one of the volatile organic compounds to detect with our electrostatic sensor because porous silicon-based IPA sensors have been reported previously. Consequently, we can easily compare our results with those of the IPA sensors in terms of the sensor performance such as the detection limit and response time. The proposed electrostatic gas sensor is similar to an electrostatic speaker in terms of the construction and working principle. Recently, Zhou et al. reported on an electrostatic speaker with a conducting graphene diaphragm and evaluated the properties of the device as an audio speaker [17]. In the speaker, a DC-biased graphene diaphragm is suspended between two stationary conductive sheets that form an alternating electric field. This causes the diaphragm to vibrate with the frequency of the electric field by electrostatic force, and the vibrating diaphragm produces sound waves. According to the Zhou et al.'s paper [17], to fabricate an electrostatic speaker to generate sounds almost exactly like the original sound, a diaphragm with a flat frequency response in the audible frequency range is required. In comparison, the electrostatic gas sensor proposed in this paper requires a diaphragm with an uneven

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http://dx.doi.org/10.1016/j.snb.2015.04.118
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frequency response caused by diaphragm resonance in the given frequency range, although the construction of the other parts is identical to the case of an electrostatic speaker. The reason for this requirement is that the electrostatic gas sensor uses the change of the vibration amplitude and the shifts in the resonance frequency of the diaphragm as response signals when in contact with the target gas. To meet this requirement, we tried to use a freestanding porous silicon film as the diaphragm of the electrostatic gas sensor. The conductivity of the porous silicon layer increases with the concentration of the target gas to be detected. Consequently, the vibration amplitude of the diaphragm in the alternating electric field can be varied with the concentration of target gas. Furthermore, the increase of the diaphragm mass by capillary condensation and absorption of the target gas may shift the resonant frequency.

The principle of gas sensing using the resonance frequency shift is similar with that of quartz crystal resonator (QCR) sensors. QCR sensors, which are coated with different polymers or are etched to produce micropores into the surface of the resonator, have been studied intensively in relation to the detection of organic vapors, humidity, and organic compounds in liquids [18–20]. According to the operation principle of these sensors, the resonant frequency of quartz crystal is shifted because the molecules of the target gas are absorbed into the coating material or the porous structure.

This study proposes the working principle of an electrostatic gas sensor with a porous silicon diaphragm and investigates its IPA gas sensing properties. To this end, the frequency response of the diaphragm was measured as a function of alternating electric field frequency. As response signals, the changes of vibration amplitude and the shift of the resonant frequency of the diaphragm when in contact with IPA gas were also investigated. Finally, the dependence of the operating voltage, which is applied to the diaphragm and to two parallel conductive sheets to produce an alternating electric field, on the vibration amplitude of the diaphragm was evaluated, and directions for future study to enhance sensor performance are discussed.

2. Experimental details

2.1. Fabrication of a porous silicon diaphragm

Porous silicon layers, having the photonic crystal structure of a rugate filter, were prepared by electrochemical etching of a p-type single crystal silicon wafer (Nilaco, 0.02 Ω cm) in an HF/ethanol solution (mixing ratio 1:2.3). The waveform of current density for etching was sinusoidal, oscillating between 15 and 50 mA/cm^2. The period of a cycle and the total number of cycles of the waveform were 7 s and 75 cycles, respectively. To generate the waveform, a programmable DC power supply (Agilent, E3634A), controlled by LabVIEW, was used.

Because a rugate-structured porous silicon layer is still attached to the silicon substrate, the layer should be separated from the substrate to obtain a freestanding porous silicon diaphragm. To do this, the interface region between the porous silicon layer and the silicon substrate was electropolished via a second etching process performed with a constant current density of 13 mA/cm^2 for 200 s in an HF/ethanol solution (mixing ratio 1:14.2).

To study the effect of annealing on the vibration properties of the diaphragm, as-prepared diaphragms were annealed at 300 °C for 1 h in an air atmosphere using an electric furnace (Lab House, DMF-3).

Fig. 1(a) shows a scanning electron microscope (SEM, TESCAN, MIRA3) image of the cross-section of the non-annealed porous silicon diaphragm. Many horizontal stripes, a unique feature of rugate-structured porous silicon, are observed in the image. These stripes are attributed to the regions etched by low and high current densities having sinusoidal waveform, respectively. Fig. 1(b) shows an SEM image of the cross-section of the annealed porous silicon diaphragm. Compared to Fig. 1(a), the strips are not observed clearly, but pore sizes have become small. This phenomenon is also seen in the surface images of the non-annealed (Fig. 1(c)) and annealed (Fig. 1(d)) porous silicon diaphragms. Numerous pores with diameters of approximately 29 nm, as shown in Fig. 1(c), have become as small as 14 nm after annealing. Therefore, thermal annealing may increase the sensitivity of the sensor by increasing the specific surface area as well as enhancing membrane stiffness by oxidation. The shape of the annealed porous silicon diaphragm is shown in Fig. 1(e). Instead of a silicon substrate, the diaphragm is placed on an aluminum sheet. To use this as a vibrating plate of an electrostatic gas sensor, the diaphragm was fixed between two polycarbonate sheets perforated with a circular hole having a diameter of about 4.5 mm, as shown in Fig. 1(f).

2.2. Fabrication of an electrostatic gas sensor

Fig. 2 shows a schematic diagram of the proposed electrostatic gas sensor. The porous silicon diaphragm was placed between two perforated polycarbonate sheets with thickness of 0.5 mm. Also, the edge of the diaphragm was connected to one end of a copper wire (MK electron, 99.99% purity) with a diameter of 25.4 μm using silver paste. The other end of the copper wire was connected to a DC power supply (Keithley, 2400 SourceMeter) to provide voltage, V_{DC}, of up to 70 V to the diaphragm. Two aluminum plates having a thickness of 0.5 mm were respectively attached to the upper and lower surface of the overlapped polycarbonate sheets. Here, the lower side plate was perforated with a rectangular array of air holes with diameters of 0.6 mm. The lattice constant of the rectangular hole array was 1.2 mm. To form an alternating electric field between the two parallel aluminum sheets, sinusoidal alternating signals generated from a function generator (Agilent, 33220A) were respectively applied to each aluminum sheet. Here, to make a phase difference of 180° between the alternating signals applied to each aluminum sheet, a signal inverter was used, as illustrated in the figure. The frequency, f, of the sinusoidal alternating signal was controlled in a range of 0.5–40 kHz, and the peak voltage, V_{AC}, of the signal was also controlled in a range from 0 to 7.0 V.

IPA gas was admitted into the vibrating diaphragm through the holes of the perforated aluminum sheet, and the sound generated from the diaphragm also passed through the holes to a condenser microphone (AKG, DS-C 1000S). According to the specification sheet, the detectable sound frequency range of the microphone used in this study was from 50 Hz to 20 kHz. However, to investigate the frequency response of the diaphragm in a wider range, we used the microphone to detect sound waves having a frequency range of 0.5–40 kHz, despite the fact that output signal distortion will occur above 20 kHz. A digital oscilloscope (Lecroy, Waverunner 6051) was used to monitor the waveform of the electric signal produced by the microphone. The output signal of the microphone was also sent to a lock-in amplifier (EG&G, 5302) to improve the signal-to-noise ratio, and then the output voltage of the amplifier was monitored as a measure of the vibration amplitude of the diaphragm, using a digital multimeter (Hewlett Packard, 34401A). The digital multimeter was connected to a personal computer by a GPIB interface (National Instruments, GPIB-USB-HS) and communication software.

3. Results and discussion

3.1. The frequency response of the diaphragm

Fig. 3 shows sound waveforms emitted by the electrostatic gas sensor with a porous silicon diaphragm at different f values of the

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