A Prototype Fabrication of Sensitive Porous Silicon NOx (x=1,2) Gas Sensor

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ABSTRACT

A prototype of (porous silicon (PS) based) NO_x(x=1.2) gas sensor based on porous silicon (PS) (operated at room temperature) has been fabricated. The sensor could be operated at room temperature. The (PS) basic material for the material sensor (i.e. PS) was fabricated in the previous research by varying (parameter of the HF concentrations) concentrations of HF (what is HF?) and (the anodization time) time of anodizations. Its structure, as well as its optic, has been characterized. In this research, we did electrical characterization of PS to find its conductivity. The conductivity of PS is important because PS, as a sensor, utilizes the changes of conductivity when it is passed by NO_x(x=1.2). (The influence of these parameters on I-V characteristics of the PS and on the sensor performances has been studied in detail) The changes of electrical current because of the varying NO_x concentrations with time were measured using a current measuring system, then plotted. The result (shows) showed that the sensor could detect NO (and NO_2) gasses ranging from 16 ppb to 200 ppb and NO_2 gas ranging from 12 ppb to 200 ppb, respectively at room temperature (28°C) and at adjusted currents ranging from 20 to 800 μA. The result also showed that the sensor was not completely reversible for NO_x.

Keywords : Porous Silicon (PS), sensor, NO_x gas

INTRODUCTION

Porous Silicon (PS) properties have been studied by (some) researchers. The studies have shown that it is possible to apply PS for (as) photo detectors (Bilyaloy *et al.* 2000), for sensitive gas sensors at low voltage sources, low cost and room temperature (Gole 2002), and for photovoltaic cells (Martin-Palma *et al.* 2004).

Porous silicon can be fabricated using Hydrofluoric (HF) solution, and When fabricated using HF based solution, its properties depend on several factors, such as substrate resistivity (Si wafer), current density, time of anodization, HF concentration, pore dimension, porosity, and material texture (Pacebutas 1996).

Nitrogen monoxide (NO) and nitrogen dioxide (NO_2) are toxic air pollutants emitted by engine combustions. Several countries have assigned a warning level for these gases at about 150 ppb. Above this level, they become very dangerous for lungs (Van Klinken 1991). Unfortunately, a sensitive but inexpensive sensor for detecting low levels of NO_x is currently unavailable, especially in Indonesia. Recently, several sensors for detecting NO_x level close to 200 ppb have been developed in the world. In this paper, we present a prototype NO_x sensor based on the conductivity changes of the PS layer. We chose one of the PS samples for the sensor based on its electrical characteristic.

METHODS

In the previous research (study), we fabricated PS by varying time of anodizations and HF concentrations (Sudiana *et al*. 2007). We used a single-crystalline Si (111) p-type wafer with a resistivity of 0.1 Ωm. A porous silicon layer was grown by an electrochemical dissolution in an of (a) HF solution. The concentrations of HF solution used were 40%, 30%, 20%, and 10%. We applied an etching current density of 50 mA/cm^2 and anodization time of 10, 20, 40 and 60 minutes for each HF concentration. After being fabricated (fabricating), PS samples were characterized using SEM micrographs in P3G Bandung to determine the pore size of PS, and using FTIR at the laboratory of Chemistry Analytic of Gadjah Mada University to identify the functional groups of SiHx (x=1,2,3). Besides (them), their structures were determined using XRD at the Mining Laboratory of Bandung Institute of Technology .

In this research (study), the best electrical properties of PS were we did electrical characterization of the PS that was then followed by choosing porous silicon having the best electrical properties for the sensor. The electrical characterization of the PS was conducted using an Electrical. Characterization of Semiconductor Material device designed by Muhtiar at the Material Physics laboratory of Haluoleo University (Muhtiar *et al*. 2007). Its (The current (I) and voltage (V) characteristics) were simultaneously measured in vacuum and non-vacuum chambers using Four probe method. After (determining) a PS for NO_x sensor had been determined, (the) metal electrodes were deposited by evaporation (evaporating) on the porous silicon's top surface. (The) copper wires were connected to electrodes using an epoxy silver paste. During measurement, the sensor was kept in a sealed chamber. NO_x gas flux from gas cylinder was adjusted to certain value to achieve concentrations ranging from 12 ppb to 200 ppb.

RESULTS AND DISCUSSION

Porous silicon characterization

The previous research (study) on the characterization of PS (Sudiana *et al.* 2007) can be described briefly as follows. The porous silicon was fabricated using 10%, 20%, 30%, dan 40% of HF solutions. From the research, the PS fabricated with (formed by) 20% of HF had the best electrical conductivity, therefore, it was chosen as a sensor. The X-Ray Diffraction (XRD) showed that the PS structure was monocrystalin at first (i.e. it has one peak). The peak peak's height then decreased with (increasing the anodization time. This decrease indicates that the structure gradually changed to amorf. This result was confirmed by the SEM photograph of the PS magnified 20.000 times. The diffraction spectrum also showed that diffraction's angle (2θ) was in agreement with that of silicon (111) p-type (i.e. about 28°). Using Bruggeman approximation, we had estimated that its porosity was about 78% (Bisi *et al.* 2000).

Moreover, using FTIR, the PS contained functional groups of SiH, SiH_2, Si-Si, and Si-O-Si. The PS samples (formed by) fabricated with 20% of HF and 10, 20, 40, and 60 minutes of anodization time are shown in Figure 2. Further, the electrical properties were analyzed. During the electrical characterization, it was found that the conductivity of the PS varied from 1.46 E-04 to 8.54 E-05 $(\Omega m)^{-1}$.

From the electrical property, the PS samples (formed by) fabricated with 20% of HF and 20 minutes of anodization was chosen as a NO_x sensor because of its stable conductivity with temperature as shown in Figure 3. The initial measurements of the PS's electrical current in open and vacuum (P=300 torr) chambers are shown in Figure 4. The current in the open chamber was greater than that of in the vacuum chamber. This is due to the fact that air contains water vapor that has conductive property so that it influenced the measured PS conductivity.

Porous silicos as NO_x sensor

Figure 5 shows the response of the PS-based NO_x sensor on the concentrations of NO ranging from 16 ppb to 200 ppb and NO_2 ranging from 12 ppb to 200 ppb at room temperature (28°C). An increase in electrical current with the increase of NO_x concentrations can be observed in Figure. 5. The graph also shows the recovery process of the sensor after being exposed to the gases. It seemed that the recovery was not completed presumably due to an irreversible interaction between the NO_x (either the NO molecules or the NO_2) and the porous silicon's internal surface. This agreed with reversible quenching effect of PS photoluminescence in the presence of NO_x (Gaburo *et al.* 2001). The reversibility of sensor process is one of the main problems in a gas sensor development.

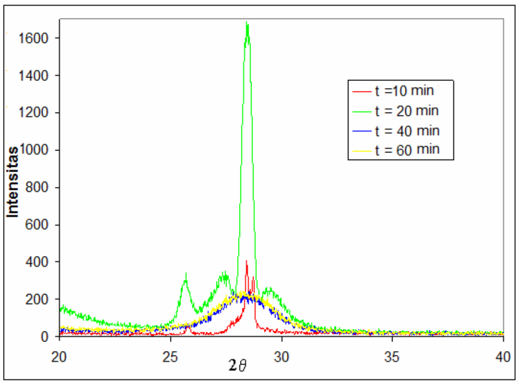

Figure 1. XRD spectrum of PS for 20% HF concentration and anodization time of 10, 20, 40 and 60 minutes.

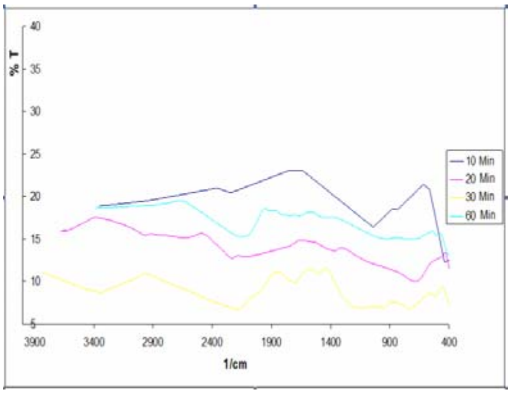

Figure 2. FTIR result of PS using 20 % of HF and 10, 20, 40, 60 minutes of anodization time.

Figure 5. also shows that the sensor did not completely recover in long time. (The

conductivity) of PS-based NO_x sensor conductivity increased (with increasing) the concentration of NO_x. when the concentration of NO_x was increased.

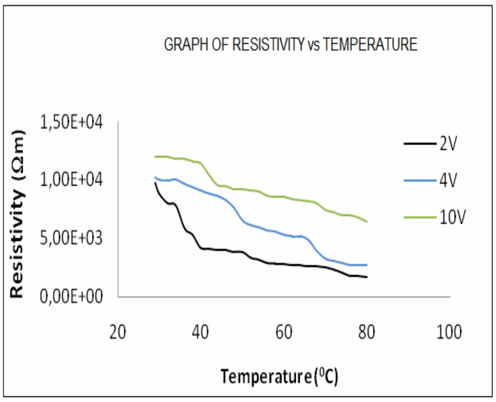

Figure 3. Resistivity vs Temperature of PS fabricated (formed by) using 20% of HF and 20 minutes of anodization time.

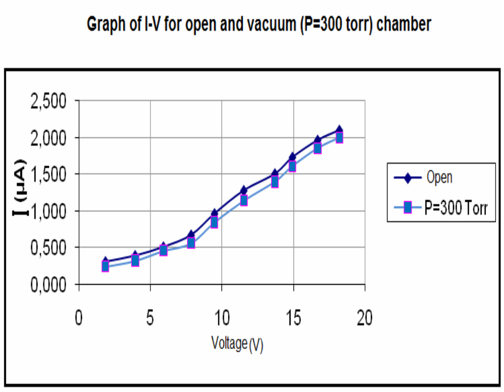

Figure 4. The *I-V* graph of PS in open and vacuum (P=300 torr) chambers.

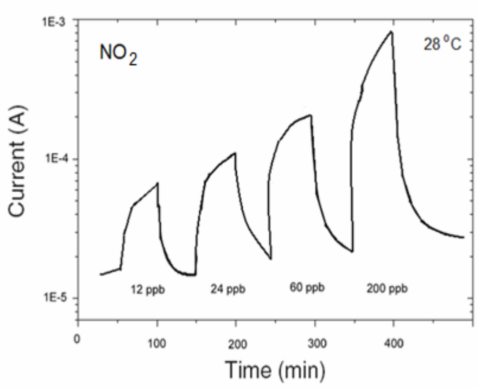

Figure 5. NO_x sensor response on NO and NO_2 gases.

This explained the interaction between the NO_x molecules and the PS when the gases were flowed on the sensor and adsorbed by the PS surface.

The NO_x molecules acted as acceptor centers that led to an increase of free carrier (i.e. holes) concentration in the PS. The increase of the carrier thus increased the conductivity of the sensor. The prototype of sensor in this research has been tested in laboratory scale only and we plan to continou this research until find a comercial sensor.

CONCLUSION

A prototype of a sensitive NO_x (x=1,2) sensor has been developed. It could detect low concentrations of NO_x at room temperature. However, the effect of NO_x on the sensor was not completely reversible.

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