



Short communication

Organic vapor detection using a color-difference image technique for distributed Bragg reflector structured porous silicon

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ARTICLE INFO

Article history:

Received 4 December 2009

Received in revised form 25 February 2010

Accepted 23 March 2010

Available online 31 March 2010

Keywords:

Porous silicon

Color-difference image

Color response signal

Gas sensors

Organic vapors

Color-difference response

ABSTRACT

To obtain an efficient response signal for organic vapors, a color-difference image technique is applied to a distributed Bragg reflector structured porous silicon (DBR PSi) sensor. The color-difference images were produced by the mathematical conversion of each pixel from surface interference color photographs of DBR PSi taken by a high-speed camera before and during exposure to an organic vapor. The sensitivity of this color-difference response signal was higher than the photoluminescence (PL) response, and the detectable concentration range was wider than the current response. Additionally, both the response and recovery times were faster than the other two responses.

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1. Introduction

In 1990, it was observed that porous silicon (PSi) under excitation with ultraviolet light emits photoluminescence (PL) in the visible region [1]. Thus, many researchers have worked to clarify the fundamental mechanism of PSi luminescence. However, the research focus in this area has begun moving toward application areas such as sensors [2–9] and drug carriers [10,11]. PSi has a large internal surface area of $\sim 200 \text{ m}^2/\text{cm}^3$ [12]; accordingly, many researchers have intensively investigated this material to use as a sensor to detect various gases [2–6], liquids [7], and biological molecules [8,9]. Moreover, the changes of the physical quantities of PSi, such as the reflectance [3,5,6], electrical conductivity [4,13], and PL [2,13], have been harnessed as response signals for target substances.

Particularly, reflectance measurements are commonly used as optical remote detection techniques as opposed to electrical conductivity measurements, which require physical electrical contact on the sample surface. In single or the multilayer PSi, a shift of the reflection spectrum due to exposure to a target substance appears due to a refractive index change in the layers, as the substance penetrates into nanometer-sized pores by micro-capillary action [5]. As a result, many researchers have reported a shift in the spe-

cific wavelengths in the reflectance spectrum as a response signal [3,5,6]. However, the reflectance spectrum shows the sum of the information of a large analysis area in a sample. Therefore, an extremely localized variation in the reflectance in a sample cannot be detected effectively.

The spectral shift corresponds to the change in the color of the sample surface. The objective of this study is to discuss the sensitivity of the change in the interference color of DBR (distributed Bragg reflector) structured PSi exposed to different concentrations of an organic vapor. DBR PSi exhibits a high reflectivity stop band due to interference in the optical reflectance spectrum. Therefore, a specific interference color corresponding to wavelengths of the stop band is observed on the DBR sample surface. Therefore, the use of a DBR PSi sample is suitable to achieve the research objective because the interference color will be changed distinctly by exposure to organic vapors.

To determine the degree of the change of the interference color, a color-difference image technique was applied based on color science in sensor research. The color difference, ΔE , is a single number that represents the distance between two colors. The value of ΔE can be formulated by the color-difference equation created by the international commission on illumination (CIE) in 1976 [14]:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

where L^* is the lightness, a^* is the position between red/magenta and green, and b^* is the position between yellow and blue in the CIE $L^*a^*b^*$ color space. Based on this theory, a color-difference

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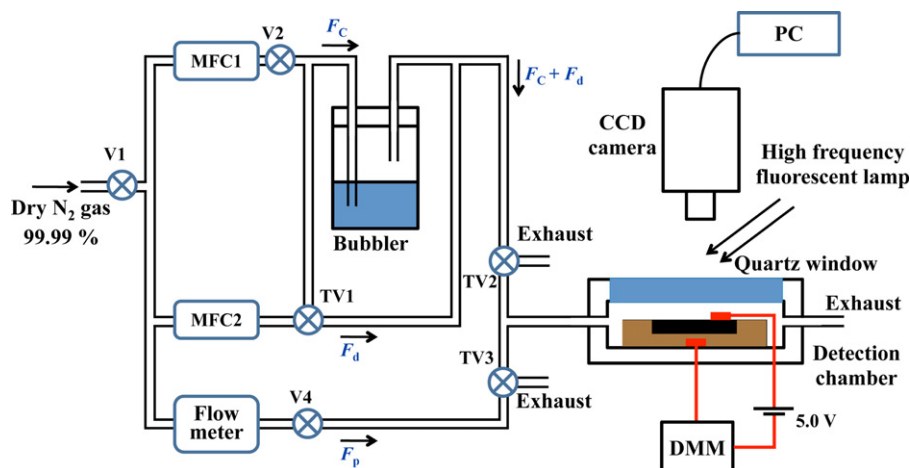


Fig. 1. Schematic diagram of the experimental system showing the valves (V), three-way valves (TV), mass flow controllers (MFC), a bubbler, a detection chamber, and the data acquisition system.

image between a reference and a sample photograph can be made if all color-difference values between the pixels that compose both photographs are computed by the aforementioned equation.

In this paper, the color-difference images of DBR PSi were successfully produced before and during exposure to an organic vapor at different concentrations. Moreover, a color response signal obtained from color-difference images was compared with conventional response signals of the PL intensity and current. To do this, the sensitivity, detectable concentration range, response and recovery times of each signal were examined.

2. Experimental

A 20-pair DBR PSi sample was prepared by the electrochemical anodization of a p-type Si (1 0 0) wafer ($0.06\text{--}0.12\ \Omega\text{ cm}$) in a 15% HF ethanolic solution. The low and high refractive index layers in the DBR were formed by anodization current densities of 100 mA/cm^2 for a time of 2.44 s and 25 mA/cm^2 for a time of 3.35 s, respectively. The prepared DBR PSi surface showed a greenish tinge, which appeared due to the interference effect of light by the multilayer structure.

Fig. 1 shows a schematic diagram of the experimental system. This system consisted of three major parts: a vapor generator, a detection chamber, and measurement system. In the vapor generator, a bubbler was utilized to vaporize pure liquid ethanol at a purity of 99.9%. The delivery system was composed of two- and three-way valves and two MFCs (mass flow controllers). As shown in Fig. 1, the carrier flow, F_c , was controlled by setting the flow rate of MFC1 (Brooks Instrument, 5850E) with a maximum flow rate of 200 sccm (standard cubic centimeters per minute). The carrier flow was mixed with the dilution flow, F_d , which was controlled by MFC2 (MK Precision, TSC-110) with a maximum flow rate of 1000 sccm. In the experiment, the total flow rate, $F_c + F_d$ which was introduced into the detection chamber was kept constant at 300 sccm. Here, the value of F_c was selected as 5, 10, 25, 50, 100, 150, 200, and 300 sccm. And the value of F_d was selected as 295, 290, 275, 250, 200, 150, 100, and 0 sccm. In the $F_c = 300$ sccm, however, the maximum flow rate of MFC1 was exceeded. Hence, in this case, $F_c = 300$ sccm was supplied wholly by MFC2 after the dilution flow was halted. To do this, valve V2 was closed and valve TV1 was opened toward the bubbler side to flow carrier gas which is outputted from MFC2 at 300 sccm. To clean the chamber environment, 2000 sccm N_2 gas as a purge gas, F_p , was introduced into the detection chamber for 3 min before the measurement.

The concentration of generated ethanol vapor was measured using detector tubes (GASTEC, 112 and 112L). The values corresponding to the different carrier flow rates were 65, 140, 800, 2000, 4500, 10,000, 15,000 and 22,500 ppm (parts per million), respectively.

The detection chamber had a small volume of approximately 7.0 cm^3 . Inlet and outlet pipes were connected to the side of the chamber along the axial direction. The front of the chamber was covered by a quartz plate to transmit fluorescent light for the acquisition of photographs and laser light for the PL intensity measurement.

To take digital photographs of the sample surface continuously during the experiment, a high-speed color CCD (charge-coupled device) camera (Allied Vision Technology, Pike F-032C) with a maximum of 208 frames/s was used. The gamma correction of this camera was turned off. To eliminate the photo noise produced by random variations of brightness or color from the lights of the camera, a high-frequency fluorescent lamp equipped a DC/AC inverter was used as a light source.

The current and PL intensity response signals were measured to compare with the color-difference response signal obtained from this research. For the measurement of the current response, electric contacts were made using silver adhesive on the top of the PSi layer and on the bottom of the silicon substrate. Bias voltage was also applied between two contacts at a constant voltage (5.0 V) by a power supply (Agilent, E3634A), and the current was measured by a digital multimeter (National Instrument, PXI-4072). The details of the circuit are shown in Fig. 1.

The measurement of the PL response was carried out separately using the 477 nm line of an Argon ion laser (Omnichrome, 543-AP-A01) as an excitation source. Experimental detail of the PL intensity measurement setup is presented in Fig. 2. To measure the PL intensity variation, another black and white CCD camera (Sony, XC-HR50) capable of a maximum of 60 frames/s was used. The camera monitored a PL spot produced by two microscope objective lenses. The captured PL spot images were analyzed automatically through image processing based on vision technology to acquire the PL intensity variation.

All experiments in this research were conducted at room temperature (approximately 20°C).

3. Results and discussion

Fig. 3 shows the photographs and color-difference images of DBR PSi. Fig. 3(a) is a photograph taken while 300 sccm N_2 gas

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