Response Characteristics of Silicon Microring Resonator Hydrogen Sensor [†]

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Abstract: A silicon microring-resonator (MRR) hydrogen sensor which utilizes platinum-loaded tungsten oxide (Pt/WO₃) thin film was fabricated and evaluated. The uniform film was deposited on MRR portion by using sol-gel technique. By the exposure to pure hydrogen gas, the sensor device showed the large resonant wavelength shift at room temperature. It is suggested that the change in the optical properties of hydrogen sensitive layer results in this response.

Keywords: microring resonator; silicon waveguide; platinum-loaded tungsten oxide; hydrogen sensor; resonant wavelength; gasochromic material

1. Introduction

When hydrogen is used as a fuel, it only emits water theoretically. And hydrogen is generated in various ways. Thus hydrogen has been expected as a future energy carrier for clean and sustainable energy society, however, we must take particular care to utilize it because it has wide combustion range (4–75 vol. %), small ignition energy (0.018 mJ), and it leaks easily because of its small molecular size. To prevent serious accidents, a hydrogen sensor which can detect less than lower explosion limit concentration has strongly been needed and many kinds of sensors have been investigated. Among them, optical sensors have explosion-proof structure because they have no electric contacts and they have immunity to electromagnetic interferences. So they could provide good reliability [1–3].

Many kinds of optical device including fiber Bragg grating [1,4], Fabry-Perot interferometer [2,5], and Mach-Zehnder interferometer [6] are utilized for chemical sensing. Among them, the microring resonator (MRR) is very small and sensitive device and has considerably been interested recently. Researchers have tried to detect chemical or bio analyte with it. Although many papers on the MRR sensor say that it can detect specific analyte in solution [7–9], there are a few papers saying that it can detect gas analyte. According to the papers, the gas analyte can be detected by catalytic combustion [10] or expansion of the sensing film on the MRR [11]. We thought that if the MRR can detect gas analyte by other method, more kinds of analyte can be detected by this, and tried to detect hydrogen using the silicon MRR and platinum-loaded tungsten trioxide (Pt/WO₃) known as the gasochromic material by the change of the optical property of Pt/WO₃ film.

2. Materials and Methods

The silicon MRR chip was fabricated by CMOS compatible process. A window was on the MRR so this part was covered with nothing (shown in Figure 1). And then, SiO₂ was sputtered on the chip as a buffer layer. The thickness of the layer was approximately 700 nm. Table 1 shows the size of the MRR.

Table 1. The size of the MRR.

Height [μm]	Width [μm]	Roundtrip Length [μm]
0.21	0.40	69.3

Figure 1. The SEM image of the MRR of the chip.

Pt/ WO_3 was fabricated by the sol-gel method. 4.5 mL of mixture solution of ethanol and distilled water (volume ratio; 8:1) and 500 μL of 0.77 M H_2PtCl_6 solution were mixed with magnetic stirrer. 5 drops of acetylene glycol surfactant were added and stirred over 30 min. And then 3.3 mL of H_2WO_4 synthesized by passing 0.5 M Na_2WO_4 solution through cation exchange resin was added and stirred rapidly. This solution was dropped on the MRR part of the chip and dried for 60 min in the desiccator at room temperature. It was calcined for 60 min in the electric furnace at 500 degrees.

The evaluation of the fabricated sensor device was demonstrated with the home-made apparatus shown in Figure 2. 81680A (Agilent Technologies Inc., Santa Clara, California, USA) was used as the light source. 86082A (Agilent) was used as the optical spectrum analyzer. PAT 9000B (Thorlabs Inc., Newton, New Jersey, USA) was used to make incident light TM mode. The gas flow path was formed with polydimethylsiloxane. Test gas was injected into the path with the micro syringe.

Figure 2. The gas sensing apparatus.

3. Results and Discussion

Figure 3 shows the result of the pure hydrogen injection into the gas flow path. The dip of each spectra is called resonant wavelength. According to the graph, it is found that the resonant wavelength got red shifted as time elapsed. After 20 min exposure of pure hydrogen, the drift of the resonant wavelength stopped. This shift was approximately 580 pm. This result was about 4 times

larger than the fabricated one without surfactant. It is surmised that surfactant reduces the surface tension of the precursor of Pt/WO₃ and covers the surface of the MRR well, and as a result of this, the MRR is more influenced by the change of the optical property of Pt/WO₃.

Figure 3. The sensor response to pure hydrogen.

Figure 4 shows the result of the air injection into the gas flow path after the pure hydrogen injection. The resonant wavelength gradually got blue shifted but did not completely get back to the initial resonant wavelength after more than 40 min. This result may be attributed not to replacing hydrogen to air completely. Therefore, some remodeling of the sensing apparatus is needed before repeatability or hydrogen concentration dependence test.

Figure 4. The sensor response to air after the exposure to pure hydrogen.

4. Conclusions

We fabricated the hydrogen sensor device with the Si MRR chip and Pt/WO₃ and tested how this device respond hydrogen. The result showed approximately 580 pm resonant wavelength shifts to pure hydrogen. From this result this device can be expected as a high sensitive hydrogen sensor. To investigate sensor characteristics of it, some remodeling of the sensor apparatus or re-examination of measuring method is immediately needed.

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