Hydrogen and Humidity Sensing Based on WGMs of Elastic Polymer Optical Microresonators

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Abstract— We report hydrogen and humidity sensor employing elastic polymer (SU-8) optical microresonators. The sensing mechanism relies on the optical whispering gallery mode (WGM) resonance shifts in the transmission spectra of these microresonators. WGMs are excited through SU-8 waveguides in which a tunable laser light is coupled from an optical fiber. Relative humidity between 0 and 65% is detected at room temperature. For hydrogen sensing, the microresonators are coated with a thin layer of palladium (Pd) metal layer to increase the sensing performance. Hydrogen gas is detected reversibly between 0.3 and 1.5%. Hydrogen gas concentrations higher than 1.5% result irreversible degradation of the devices because of a phase transition occurring in the Pd layer.

1. INTRODUCTION

Hydrogen gas attracts a lot of attention as energy source courtesy of high efficiency in energy conversion and non-toxic side products [1, 2]. However, low flammable limit (4%), small molecular volume and high diffusion coefficient of hydrogen gas (H_2) introduce difficulties in hydrogen storage. Different sensing mechanisms, such as thermal [3], electrical [4], mechanical [5], acoustic [6] and optical methods [7] are used for H_2 detection. Optical sensors are generally more suitable for reversible detection of H_2 at low concentrations. First reported optical H_2 sensor relies on interferometric changes of a laser light in a Pd-coated optical fiber [8]. Afterwards, optical H_2 sensors are demonstrated utilizing reflection spectroscopy [9], reflectivity [10] or surface plasmon resonance changes [11].

In this paper, we report H_2 detection using whispering gallery mode (WGM) resonance shifts of optical microresonators fabricated by standard UV photolithography. WGM resonances of the microresonators are very sensitive to the size of the microresonator. In order to employ this phenomenon, a thin layer of palladium (Pd) is coated on the microresonators, since the lattice constant of Pd lattice increases when H_2 is adsorbed in Pd [12]. SU-8 polymer is used as the photoresist for the material of the microresonators since SU-8 is elastic and its refractive index is high enough to confine the laser light. In the literature, elastic nature of SU-8 is used for strain sensing [13], photoacoustic microscopy [14] and opto-mechanically tunable lasing [15]. Similar microresonator-based detection of H_2 down to 0.7% employs WGM shifts due to a local temperature increase because of the combustion of H_2 [16]. In another work, vertical cavity lasers are used for H_2 detection, in which the change in the complex refractive index of Pd results a shift in the lasing wavelength [17]. As compared to these previous studies, our system provides lower detection limit of 0.3% H_2 using a relatively simple fabrication technique.

2. MICROFABRICATION

Fabrication of sensor devices consists of a two-step UV photolithography procedure. In the first step, SU-8 microresonator and waveguide structures are fabricated on a thick-oxide wafer. Extra thick oxide layer provides smaller refractive index (1.445) than SU-8 (1.573) so that the light can be confined in the SU-8 layer [18]. Then in the second step Pd microdisks are deposited on these microresonators using lift-off technique. The optical power circulates at the outermost edge of the microresonator. Therefore the diameters of Pd microdisks are designed smaller than the diameters of the corresponding microresonators to avoid optical loss due to Pd metal.

3. MEASUREMENTS

Tunable laser light is coupled from an optical fiber to SU-8 waveguide using butt-coupling method [19] (See Figure 1). As the wavelength of the laser is changed in a controlled manner, the transmission spectrum is collected using another optical fiber butt-coupled to the other end of the SU-8 waveguide. Before H_2 sensing experiments, the humidity in the sample chamber is removed with dry nitrogen gas and during that time blue-shift is observed [20]. Then H_2 concentration is changed in the sample chamber using gas flow controllers and the WGM resonances are monitored. Detection signal obtained from Pd-coated and uncoated devices are given in Figure 2. 10% H_2 results about 15 pm of resonance shift in the case of uncoated microresonator. On the other hand, 15 pm resonance shift is achieved under 0.5% H_2 when Pd-coated microresonator is used. Therefore Pd coating improves the sensitivity of the device 20-fold.

H_2 detection performance of the devices are tested in the humid environment. The humidity of

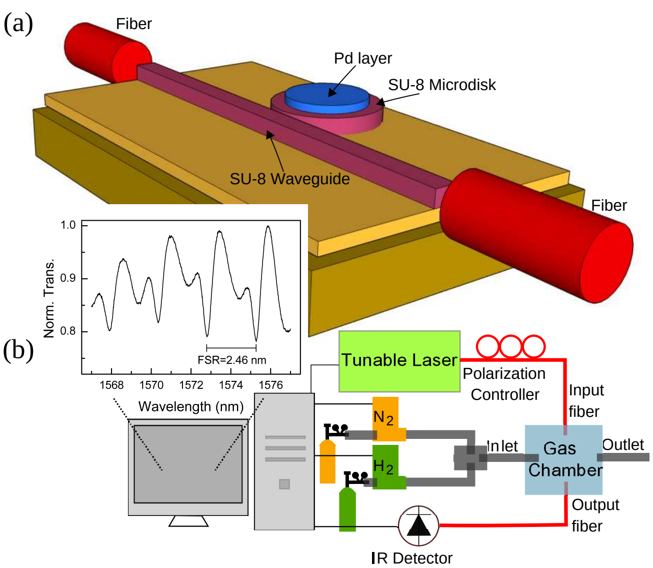

Figure 1: H_2 sensing results obtained from (a) sketch of the sample area. Tunable laser light is coupled from an optical fiber to the SU-8 waveguide. Then the optical WGMs of the Pd-coated SU-8 microresonator are excited from the SU-8 waveguide, (b) the whole experimental setup. The sample is put into a gas chamber in which the H_2 concentration and relative humidity can be changed in a controlled manner. The inset shows an examplary spectrum obtained from a Pd-coated microresonator.

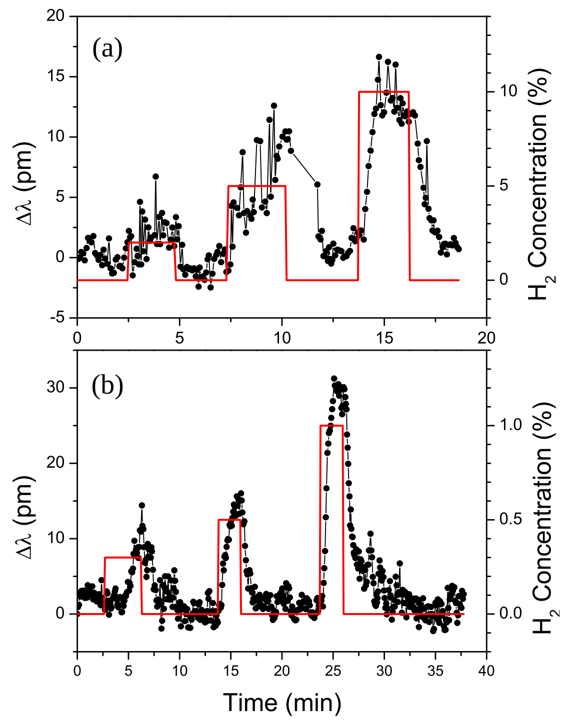

Figure 2: H_2 sensing results obtained from (a) Uncoated SU-8 microresonator and (b) Pd-coated SU-8 microresonator. Red lines indicate the H_2 concentration in the sample chamber and black dots indicate the resonance shifts.

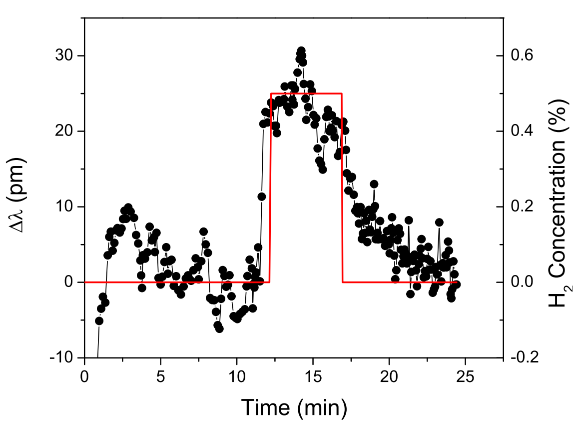

Figure 3: H_2 detection signal obtained from Pd-coated SU-8 microresonator in the presence of 51% RH.

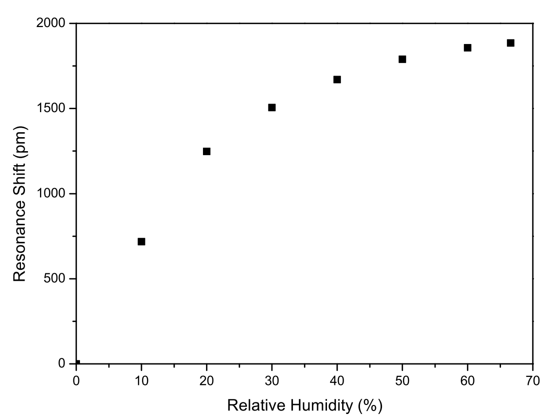

Figure 4: Humidity detection using uncoated SU-8 microresonator. Resonance shift increases as the RH increases up to 65%.

the sample chamber is kept constant at 51% relative humidity (RH) at room temperature during the sensing experiment. 0.5% H_2 is detected under humid conditions (See Figure 3). Ability to detect H_2 under humidity may lead to practical applications.

Sensing performance of the uncoated resonators are tested for humidity. In general, polymers are good absorbers of humidity [20]. Therefore there is no need to coat the SU-8 polymer microresonator with an extra active layer. This makes the fabrication procedure a lot easier. At room temperature, RH between 0 and 65% is successfully detected using transmission spectra of uncoated microresonators (See Figure 4).

4. CONCLUSION

In conclusion, H_2 sensing under dry environment and humid environment are presented as well as the humidity sensing using optical WGM resonance shifts. It is possible to reversibly detect less than 1% H_2 with or without the humidity in the environment. Pd is used as an active layer for H_2 detection to improve the sensitivity. On the other hand, humidity sensing does not require any extra coating because the material of the microresonator itself is polymer which has high absorption of humidity. At room temperature, RH between 0 and 65% is detected using uncoated microresonators.

ACKNOWLEDGMENT

This work is supported by Türkiye Bilimsel ve Teknolojik Araştırma Kurumu (TÜBİTAK) Grant No. 110T803. One of the authors, SA, thanks Co-founded Brain Circulation Scheme of TÜBİTAK for support.

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