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Optical sensor for hydrogen gas based on a palladium-coated polymer microresonator

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ABSTRACT

We report an integrated optical sensor of hydrogen (H_2) gas employing an SU-8 polymer microdisk resonator coated with a palladium (Pd) layer and coupled to a single-mode optical waveguide. The sensing mechanism relies on the expansion in the Pd lattice due to palladium hydride formation in the presence of H_2. Strain induced in the microresonator then causes a red shift of the spectral positions of the resonator whispering gallery modes (WGMs) which is monitored using a tunable laser coupled to the waveguide. H_2 concentrations below the flammable limit (4%) down to 0.3% could be detected in nitrogen atmosphere at room temperature. For H_2 concentrations between 0.3 and 1%, WGM spectral positions shifted linearly with H_2 concentration at a rate of 32 pm/% H_2. Average response time of the devices was measured to be 50 s for 1% H_2. The proposed device concept can also be used to detect different chemical gases by using appropriate sensing layers.

1. Introduction

Thanks to its high energy conversion efficiency, non-toxic side products, and sustainable nature, hydrogen (H_2) attracts a lot of attention as an energy source [1,2]. H_2 is also widely employed in clinical, industrial, and environmental reservation applications. Main difficulties in using H_2 stem from its low flammable limit (4%) and small molecular volume which bring challenges in H_2 storage and sensing. To date, H_2 sensors have been demonstrated using different measurement methods such as thermal [3], electrical [4], mechanical [5], acoustic [6] or optical [7]. Among these, optical sensing techniques are generally convenient for achieving reversible sensing with low cost and compact devices. The first optical H_2 sensor relied on the interferometric detection of the length of a palladium (Pd)-coated optical fiber [8]. This work was followed by other optical H_2 sensor demonstrations including those employing reflection spectroscopy [9], reflectivity [10] or surface plasmon resonance phenomenon [11].

In this paper, we report a novel optical sensor of H_2 gas that employs a polymer microdisk microresonator coated with a palladium (Pd) sensing layer and optically coupled to a single-mode polymer waveguide. Microdisk cavities host high quality optical resonances called whispering gallery modes (WGMs) whose spectral positions are very sensitive reporters of the microresonator size and refractive index [12]. Our sensor detects the H_2 gas via monitoring the spectral position of the WGMs that changes due to the strain induced in the microresonator as a result of expansion in the Pd sensing layer upon the formation of palladium hydride. To determine the WGM positions, we employ a tunable laser coupled into the polymer waveguide. We report H_2 detection below the flammable limit down to 0.3%, with a linear response of the sensor in the H_2 concentration range between 0.3 and 1% and a typical response time on the order of 1 min for 1% H_2.

Up to now, elastic nature of polymer microring or microdisk microresonators has been exploited in numerous demonstrations such as strain sensing [13], ultrasonic detection for photoacoustic microscopy [14], and opto-mechanically tunable lasing [15]. Despite these, H_2 gas sensing using a polymer microring or microdisk microresonator has not been demonstrated yet. Solid silicon-on-insulator (SOI) ring resonators have been previously used for H_2 gas sensing [16]. In these experiments, H_2 was detected by monitoring the spectral change in WGMs caused by the local

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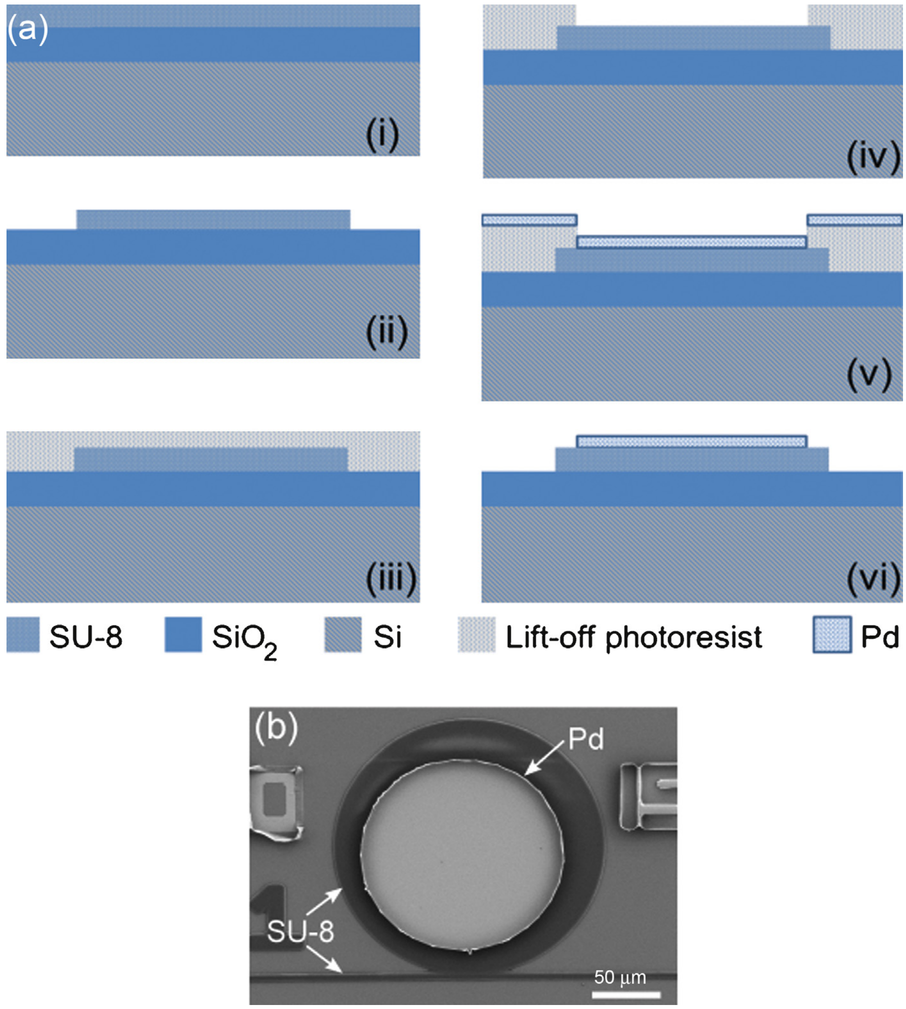

Fig. 1. (a) Cross-sectional schematic views showing the microfabrication steps of the sensor device. Thicknesses: Pd – 220 nm, SU-8 – 1200 nm, SiO_2 – 5 μm, Si – 500 μm. (b) SEM image of a 200-μm diameter Pd-coated SU-8 microresonator with SU-8 waveguide.

temperature increase resulting from the catalytic combustion of H_2. However, this approach did not allow measurement of H_2 concentrations lower than 0.7%. Vertical cavity lasers [17] were also used in microresonator-based H_2 gas sensing demonstrations. Here, H_2 gas sensing relied on the change of the complex refractive index of a Pd layer, leading to a spectral shift of the lasing wavelength. Compared to these previous demonstrations, our sensor combines easy fabrication together with the detection of relatively low H_2 concentrations (0.3%).

2. Experimental

2.1. Microfabrication

Two-step UV photolithography was used for the fabrication of the sensor devices (Fig. 1a). In the first step, SU-8 microresonators and waveguides were fabricated on a Si wafer with a 5 μm thick oxide layer using standard UV photolithography. The thicknesses of the microresonators and waveguides were measured as 1.2 μm. In the second step, a thin layer (thickness: 220 nm) of Pd was coated on the microresonators using RF plasma sputtering. Undesired portions of the Pd coating were removed using lift-off technique. The crystallographic properties of the Pd film were analyzed by X-ray diffraction (XRD) on a Rigaku Smarlab diffractometer (using Cu $K\alpha$ = 1.5418 Å radiation) in the range of 10–90°. A Zeiss Ultra Plus field emission scanning electron microscope was used for SEM imaging.

Measurements reported in this paper employed disk-shaped microresonators with or without a hollow core as shown in Figs. 1b and 7. For the cases with a hollow core, the inner diameter was between 100 and 160 μm, substantially smaller than the 200 μm outer diameter of the microdisks. Hence, the optical properties of the WGMs were not affected by the presence of the hollow core. No considerable difference was observed between the sensing performance of the microdisk devices with or without a hollow core. For different microdisks, the diameter of the Pd-coated region was selected between 150 and 176 μm, smaller than the outer diameter (200 μm) to avoid absorption and scattering of WGMs due to the Pd layer (see Fig. 1b). We also made sure that the diameter of the Pd-coated region was larger than the inner diameter (100–160 μm) for the hollow core microdisks. Specific dimensions of the five sensors studied in this paper are: Sensor 1 – inner diameter = 160 μm, outer diameter = 200 μm, Pd-layer diameter = 176 μm; Sensor 2 – inner diameter = 100 μm, outer diameter = 200 μm, Pd-layer diameter = 150 μm; Sensor 3 – inner diameter = 0 μm, outer diameter = 200 μm, Pd-layer diameter = 176 μm; Sensor 4 – inner diameter = 160 μm, outer diameter = 200 μm, Pd-layer diameter = 176 μm; Sensor 5 – inner diameter = 100 μm, outer diameter = 200 μmi, Pd-layer diameter = 150 μm.