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An integrated optical hydrogen sensor on a silicon-on-insulator platform: Effects of palladium film thickness

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ABSTRACT

In this paper, we present the design and experimental demonstration of a silicon-on-insulator based optical hydrogen sensor. We analyze the response time, hysteresis and sensitivity of our device for various palladium film thicknesses. For a 1-nm thick palladium film the response time is approximately 6 s and the sensor suffers little from hysteresis effect. These attractive features make the sensor highly useful for a wide range of applications.

1. Introduction

The high price and volatile supply source of oil as well as the desire to move towards greener sources of energy have created a growing interest in the use of alternative fuels, such as hydrogen. The global hydrogen and fuel cell market is poised to be worth $8.5 billion by 2016 [1]. As hydrogen gas is odorless, colorless, tasteless, and can be explosive in volumetric quantities as low as 4% [2], it is vital to have an inexpensive device that can quickly, reliably and safely monitor hydrogen concentrations in case there is a leak. Optical hydrogen sensing offers a number of advantages over competing technologies including compact size, increased safety and immunity from electromagnetic interference. Most optical hydrogen sensors reported to date are based on optical fibers [3–7]. Silicon-on-insulator (SOI) is an excellent platform for implementation of optical sensors, since SOI based optical waveguides are very compact and are easy to mass produce using CMOS fabrication technology. In addition, SOI has the potential to integrate electronics and photonics on the same platform. Many different sensing channels can be integrated on a single SOI chip in order to create an optical nose that would be able to detect multiple environmental variables simultaneously. Despite all the advantages of SOI waveguides for hydrogen sensing, there have been only few reports on SOI-based hydrogen sensor [8–11] and many important questions remain unanswered. One such issue is the effect of Pd thickness on sensor performance. It has been reported for various types of hydrogen sensors that the thickness of palladium (Pd) film has a significant impact on the performance of the sensor including its response time, sensitivity and hysteresis [12]. The aim of the present work is to present a detailed analysis of SOI-based hydrogen sensor with an emphasis on the role of Pd thickness on sensor performance. Such an analysis will enable one to better assess the usefulness of these sensors for practical applications. The paper is organized as follows. In Section 2, we briefly describe our sensor design, fabrication methods and the experimental setup used for sensor characterization. We fabricated a series of sensors with various Pd thicknesses. Test results for these sensors are presented in Section 3. From our experimental results, we observed a number of trends in sensor performance. We discuss these trends, and provide some physical explanation causing these trends in the same section. We conclude the paper with a summary of our observations in Section 4.

2. Materials and methods

2.1. Principle of operation and implementation of sensor

A schematic of the hydrogen sensor is shown in Fig. 1(a). It consists of a rib waveguide partially covered by a thin Pd film. The waveguide mode interacts with the Pd layer as it propagates along the waveguide. When the sensor is exposed to hydrogen, Pd

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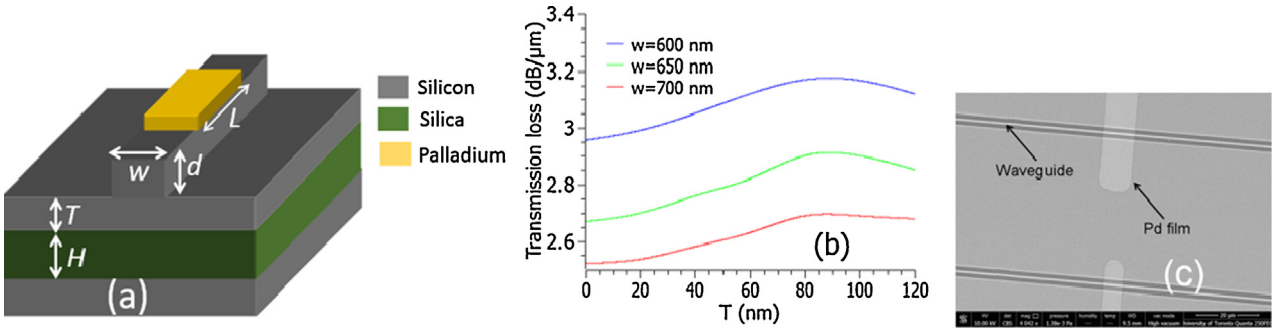

Fig. 1. (a) Schematic of the hydrogen sensor, (b) simulation results showing transmission loss as a function of waveguide dimensions for SOI rib waveguide coated with 10-nm thick Pd film and (c) SEM image of fabricated hydrogen sensors.

becomes palladium hydride, and there is an associated change in the complex permittivity of the metal film. The degree of change of Pd film depends on the concentration of hydrogen. Therefore, by measuring the change in transmission, one can estimate the hydrogen concentration. The sensor is fabricated on a SOI substrate, which has a 220-nm thick waveguide layer separated from the silicon substrate by a 3-μm thick silica lower cladding layer. We chose to use single-mode, rib waveguides for our sensor design, since the mode size is larger for these guides compared to silicon nanowire waveguides, and coupling to fiber is more efficient. To ensure that the sensor is compact, the waveguide dimensions were designed to ensure that light transmitted through the sensor strongly interacts with the Pd film, i.e. light should experience large attenuation, while propagating through the sensor. Fig. 1(b) shows the transmission loss calculated by lumerical mode solution for the transverse electric (TE) polarization for various choices of waveguide dimensions when the sensor is coated with a 10-nm thick Pd film. The plot shows that the sensor provides strong interaction with the metal for a wide range of dimensions, and the performance of the sensor is tolerant to fabrication imperfections. In these simulations we assumed that the metal is deposited only on top of the waveguide, and not on the sidewalls. This is a good assumption as the electron beam evaporation process will result in minimal side wall deposition for a thin metal film. In the presence of the metal coating on the sidewalls the transmission loss will be different than those reported in Fig. 1(b), but the device acts as an effective hydrogen sensor in both cases. More details about the sensor design can be found in [13].

The silicon waveguide was fabricated using a combination of electron beam lithography, reactive ion etching and lift-off to produce the final device. The fabrication started with spinning positive electron beam resist ZEP520 on the silicon wafer. The waveguide patterns were defined with a Vistec EBPG5000+ Electron Beam Lithography System in a Class 100 cleanroom. After the pattern was developed, reactive ion etching, using SF_6 and O_2 was used to complete the waveguide fabrication. For the lift-off process, a metalized mask and UV photolithography were used to expose the areas of the waveguides where the Pd was to be deposited. Electron beam evaporation was used to deposit different thicknesses of Pd on the sample. The Pd film was deposited using a BOC Edwards Auto 306 Electron Beam Evaporator equipped with a quartz crystal thickness monitor for real time monitoring of metal thickness. The thickness monitor needs to be properly calibrated to ensure accurate thickness measurement. We prepared a number of samples with various Pd film thickness, and compared the thicknesses predicted by the thickness monitor to those measured by an atomic force microscope. We then used the calibrated thickness monitor to estimate the Pd film thicknesses of the devices we tested. Finally, lift-off was performed to selectively remove the unwanted Pd. Fig. 1(c) shows the final devices.

2.2. Experimental setup

A diagram of the complete setup used for testing the hydrogen sensor is shown in Fig. 2. Light from a Thorlabs S5FC superluminescent diode with 1531.6 nm center wavelength, 60 nm bandwidth, and 21 mW output power was amplified by a JDS Erbium-doped fiber amplifier, and coupled in and out of the sensor using Newport 60× objectives lenses. A fiber polarization controller, polarization beam cube and a half-wave plate were used to control the input polarization. The end fire coupling rig supporting the sample and objective lenses were covered by a polycarbonate box of dimensions 38.5 cm × 18.5 cm × 12.5 cm. Germanium photo detectors were used to monitor the input and output power levels. To minimize the effects of noise, Stanford Research Systems SR830 lock-in amplifiers were used for both input and output signal detection. An optical chopper was placed in the setup after the alignment mirrors to modulate the input light at a frequency of 364 Hz. Although the guide supports both TE and TM modes, the TM mode in high index contrast ridge waveguides are inherently leaky [14]. For this reason, only the TE mode was used in our experiment. We tested the response of our sensor under exposure to various hydrogen concentrations ranging from 0% to 4%. Fig. 2 (left side) shows the set up for controlling the gas composition. Two cylinders containing pure nitrogen and 4% hydrogen (balanced by 96% nitrogen) were connected to a T-junction through two Brooks 5850S mass flow controllers (MFC). A Brooks 0154 control unit was used to control the flow rate of both MFCs. The output of each individual MFC led to a T-junction to mix the gases to achieve the desired hydrogen concentration. The output of the T-junction then led to a union connection on the box. A stainless steel tube then directed the gas flow directly onto the sample, with a sample to tube separation of about 5 mm. A commercial hydrogen sensor from Nova Analytical Systems was used to verify the hydrogen gas concentration in the gas mixture. A custom LabVIEW program was used for both controlling the hydrogen concentration and data acquisition during the experiment.

3. Results and discussions

The most important characteristics of a hydrogen sensor are sensitivity, response time and reproducibility of results under repeated exposure to hydrogen. In the following sections we describe the characterization of the optical hydrogen sensor.

3.1. Temporal response

The temporal detection response experiments were carried out by first exposing a sample to a constant flow of pure nitrogen gas to establish a baseline response. Next, the gas flow was changed from pure nitrogen to 4% hydrogen (with a balance of nitrogen).