

Integrated optical gas sensors on silicon-on-insulator platform

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Gas sensors find significantly important applications in areas such as industrial process and safety control, environmental monitoring, and medical breath analysis. Silicon-on-insulator (SOI) optical structures provide a very promising platform to realize compact, inexpensive, and environmentally robust integrated gas sensors. We demonstrate highly sensitive micro-optical hydrogen and ethanol sensors on SOI microring resonators (MRR) using chemical coatings sensitive to the corresponding gases. In the hydrogen sensor, the MRR resonance shifts to longer wavelengths due to the thermo-optic effect from the catalytic combustion of hydrogen on the sensitive coating. Whereas in the ethanol sensor, the MRR resonance shift is dictated by the evanescent field interaction with a sensitive coating whose refractive index changes upon exposure to ethanol.

Introduction

The need for detecting gases in a surrounding has been there for years for various reasons. Environmental pollution monitoring is one of the main areas where gas sensing has been playing an important role. Gas sensors have also been widely used in industrial process monitoring and leak detection of explosive gases. Another class of application for gas sensors is medical and forensic breath analysis. The concentration of some gases, such as NO, from the exhaled air of patients has been used for medical diagnosis. On the other hand, blood alcohol level monitoring from the breath of drunk drivers is an example of gas sensors for forensic applications.

Various techniques have been employed to realize gas sensors. Electrochemical, metal oxide semiconductor (MOS) and optical gas sensing techniques are the widely used ones. In recent years, the interest for optical gas sensors has been growing for several reasons. Robustness to operate in harsh environments, immunity from EM interference, compatibility to fiber networks for multiplexing and remote monitoring, low power consumption, and safety are among the attractive features about the optical sensors. Most of the optical sensors reported to date have been based on optical fibers. Fiber optical sensors have enabled multiplexed multipoint gas sensing and remote gas sensing. However, in spite of having to possess most of the advantages featured by the optical sensors, fiber optical sensors are not convenient for integration.

On the other hand, inexpensive and compact gas sensors which can elegantly fit to a wide range of applications have been of a significant interest in the gas sensing community. The possibility to miniaturize gas sensors opens a way to a chip level implementation, integration with other vital functionalities, and mass fabrication. This way, fairly cheap, highly compact and multi-purpose gas sensors can be available. However, the implementation of optical gas sensors at integrated level has been challenging mainly due to the technological limitations.

Fortunately enough, optical structures fabricated on the silicon on insulator (SOI) platform have recently been proving to be promising for a wide range of integrated optical applications [1]. These structures have been demonstrated with submicron scale features and can be realized on a very small area on a chip owing to the high index contrast between the waveguides and the surrounding claddings. Moreover, the compatibility of the SOI devices to CMOS fabrication tools and the promised inexpensive mass fabrication makes them highly attractive. Owing to these facts, the research on SOI optical structures is recently extending beyond the telecom to other applications such as optical bio-molecule and gas sensing. We demonstrate two highly sensitive gas sensors, namely, a hydrogen sensor and an ethanol sensor based on SOI microring resonators.

Integrated optical gas sensing with SOI circuits coated with mesoporous films

The indirect optical gas sensing technique, in which sensitive chemical coatings are used, readily lends itself to compact implementation compared to the direct spectroscopic sensing. Very long gas-light interaction length is usually required in the direct gas spectroscopy due to the dispersed nature of the gas molecules. The use of chemical coatings, which change their properties with the gas concentration, significantly reduces the required interaction length. Upon adsorption of the gas molecules, the coating experiences physical changes which will in turn influence the light in the underlying circuit

Highly compact SOI optical structures coated with gas specific chemical layers are very promising for integrated gas sensing. The sensitivity of such sensors can significantly be enhanced by using porous chemical coatings. Unlike dense films, in which the interaction with the incoming gas molecules is limited to the exterior surface, the porous materials provide a large accessible surface area for the interaction with the gas molecules. Accordingly, enhanced response, and hence, high sensitivity to gases is possible with porous coatings.

We have been recently working on gas sensors based on SOI optical circuits coated with mesoporous metal oxide films. Metal oxide chemical coatings have been extensively studied for gas sensing in electrical domain [2]. However, not much work has been done on their suitability for the optical gas sensing. Generally, metal oxides are transparent in VIS- NIR range. Micro ring resonators (MRRs), in particular, have been at the center of our research. Sensing with a resonance shift of a MRR lends itself to multiplexing and is less sensitive to noise from input power fluctuations.

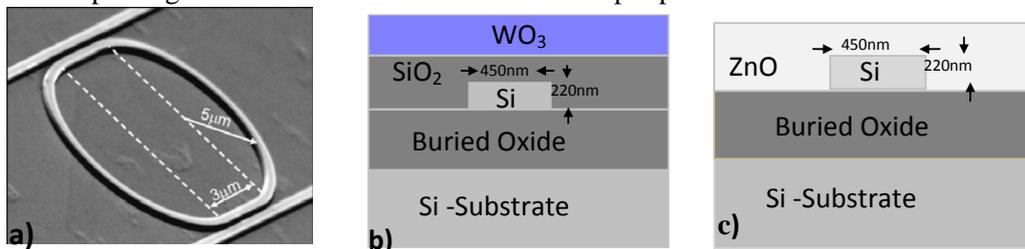


Fig 1 (a) SEM micrograph of a microring resonator, (b) & (c) cross section view of the hydrogen and ethanol sensing structure respectively.

We demonstrate two micro- optical gas sensors on SOI ring resonators with metal oxide coatings. Figure 1 shows the SEM image of a microring resonator and the basic structures of the two sensors. The first one is a hydrogen sensor; a Pt doped WO_3 catalytic film is coated on a silica clad MRR of $5\mu\text{m}$ radius. The catalytic combustion of hydrogen in an air environment heats up the underlying MRR. Due to the high thermo-optic coefficient of silicon, this temperature rise leads to a significant effective index change of the guided mode.

The second sensor we demonstrate is an ethanol sensor. Colloidal ZnO nanoparticles have been synthesized and coated to form transparent mesoporous sensitive films on SOI MRRs. The formation of porous thin films from colloidal suspensions of nanoparticles through spin or drop coating is a relatively handy technique. In this sensing technique, the sensitive coating directly interacts with the evanescent field from the underlying optical circuit. Due to the porous nature, the ethanol molecules are physisorbed at ZnO sites on a much larger surface of the film. The resulting change in the refractive index of the film shifts the MRR resonance wavelength through the evanescent field interaction.

Sample Fabrication

The microring resonator structures are fabricated by patterning and etching a 220nm thick Si top layer on an SOI wafer using 193nm deep Ultra-Violet optical lithography in a standard CMOS fabrication process [1]. The waveguide structures are made to have the lateral cross section of 450nm to achieve a Single TE mode operation. The buried SiO_2 layer has a thickness of $2\mu\text{m}$. Grating couplers, which selectively couple TE modes from an optical fiber, are fabricated at the in-coupling and out-coupling sides of the bus waveguides. The fabricated race track ring resonators have $5\mu\text{m}$ radius with free spectral range of about 15nm. The measured Q factor is over 25, 000. Such a high Q factor allows resolving very small resonance shifts down to 60pm.

The WO_3 hydrogen sensitive material is prepared using the sol-gel method, and a layer of a few microns is drop coated on the top SiO_2 cladding [3].

The ethanolic ZnO nanoparticle suspension used for making the sensitive film is prepared through a low temperature synthesis technique [4]. An nLof AZ 2070 negative photoresist patterns on microring resonators are prepared using 365nm optical lithography. 100- 200ul of the ZnO solution is drop coated on the patterned sample and left in air for about 20 minutes to dry. The photoresist is finally removed by using standard lift-off in an NMP solution.

Results

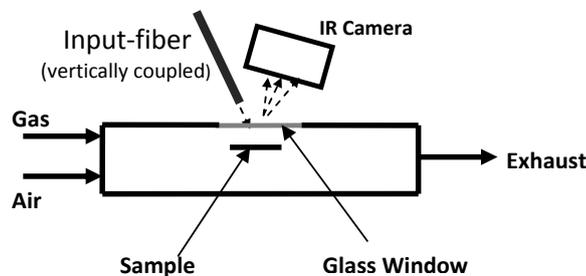


Fig 2 measurement setup

Figure 2 shows the measurement setup used for characterizing our sensors. The sensing samples are kept in a gas chamber sealed by a transparent glass window. The chamber contains two gas inlets for the gas to be measured and for the carrier gas. The exhaust at the other side of the chamber ensures continuous one way gas flow. An infrared light from a tunable laser is coupled to the sensor through the glass window by aligning an optical fiber vertically to a grating coupler on the chip. The light from the output grating couplers is collected by an infrared camera focused through the glass window.

A slight heating above the room temperature is required for the hydrogen sensor to facilitate the exothermic reaction between the hydrogen and oxygen in the air. Heating to 40 degrees is achieved by shining a light from an ordinary halogen lamp. The ethanol sensor is operated at room temperature.

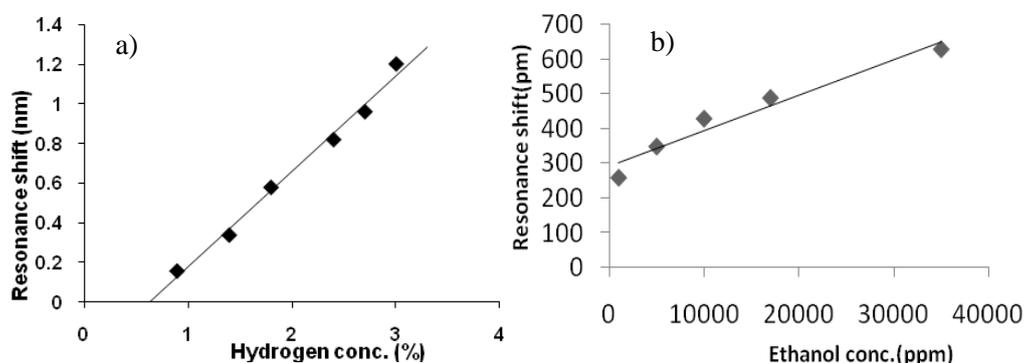


Fig 3a) measured response from the hydrogen sensor, b) measured response from the ethanol sensor

Figures 3(a) and (b) show the experimental results obtained from the hydrogen and ethanol sensors, respectively. Resonance wavelength shifts higher than one nanometer are achieved for hydrogen concentrations below the LEL. A fairly linear resonance shift of around 480pm per %H₂ within an accuracy of +/-60pm is achieved. More noticeably, a 1.2nm resonance shift is measured for 3% hydrogen in air.

High sensitivity to ethanol vapors has been achieved from the ethanol sensor. Ethanol concentrations below 1000ppm have been detected.

Conclusions

Optical structures fabricated on a CMOS compatible SOI technology are highly promising for integrated gas sensor implementation. With the aid of gas selective mesoporous chemical coatings on the SOI circuits, compact and very sensitive gas sensors can be realized on an optical chip. Using this technique, we have achieved highly sensitive integrated optical gas sensors, namely a hydrogen sensor and an ethanol sensor, based on SOI micro-ring resonators.

References:

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