

Title:

WAVEGUIDE ZEEMAN INTERFEROMETRY FOR THIN-FILM
CHEMICAL SENSORS

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Waveguide Zeeman Interferometry for Thin-Film Chemical Sensors

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Abstract

Chemical sensor based on Si_3N_4 waveguides, species-selective thin-films and Zeeman interferometry is demonstrated. Relative phase change between TE and TM modes is measured. Real-time and reversible response to toluene is shown with ppm level sensitivity.

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Introduction

Several fiber optic and integrated optics transducers have been proposed for chemical sensors [1,2]. Typically, the evanescent wave scheme is employed. The sensing is based on a change of the effective index of the guided mode due to an index and/or thickness change of a sensitive overlayer. The highest sensitivity is achieved with thin waveguides having high index differences. This yields to a vertically tightly confined waveguide mode and maximizes the portion of the energy of the mode in the thin sensitive overlayer [2].

In this letter, we propose and demonstrate thin-film chemical sensors based on silicon nitride (Si_3N_4) optical waveguides coated with the species-selective films and using Zeeman interferometry [4] as the detection technique. These highly specific surface coatings guarantee sensor selectivity while the waveguide Zeeman interferometry provides the sensitivity and stability needed for extremely sensitive detection. The species-selective thin film microsensors are based on surface modification techniques (molecular "self assembly") that allow the covalent attachment of species-selective host reagents to the surface of a sensing transducer [3]. The reagents are "bucket" like molecules that are deposited as thin, densely packed films to allow for the recognition of specific chemical compounds. These host reagents form inclusion complexes with target molecules where the molecule to be sensed fits inside the hydrophobic cavity of the host reagent. Because the formation of inclusion complexes is reversible, these reagents provide for real-time sensing of chemical agents. Sensor sensitivity can be tuned by varying the size of the host reagent cavity and by optimizing the chemical functionality of these bucket like molecules.

Our waveguide Zeeman interferometry is essentially a differential polarimetric detection technique [2], which offers the advantage that both arms of the interferometer share the same path. This features low sensitivity to common mode effects caused by environmental variations such as temperature and pressure. The Si_3N_4 waveguides on oxidized Si are ideal for this type of chemical sensors due to the tight vertical mode confinement. To demonstrate our approach we employ a monolayer cyclodextrin self-assembled film that is sensitive to toluene.

Sensor principle

Figure 1a depicts our device schematically. Light from a two-frequency Zeeman effect HeNe laser ($\lambda = 632.8 \text{ nm}$) is coupled into a Si_3N_4 rib waveguide coated with a species-selective monolayer. Our approach using the waveguide Zeeman interferometry employs the two orthogonally polarized modes generated by the Zeeman laser. These modes differ by only $3.3 \times 10^{-7} \text{ nm}$ in wavelength, i.e., 250 kHz in frequency. As these TE and TM modes propagate through the waveguide a phase difference between them is accumulated. The relative phase

difference between the TE and TM modes changes under exposure to the chemical agent, since the Si_3N_4 waveguide is highly birefringent, and the effective refractive indices of the TE and TM modes change unequally. Light from the waveguide output is led to a detector through an analyzing polarizer with transmission axis at 45° . The two laser lines interfere at the detector and produce a 250-kHz beat frequency. The phase of this 250-kHz sine wave signal is measured relative to a 250-kHz reference sine wave generated by the Zeeman laser.

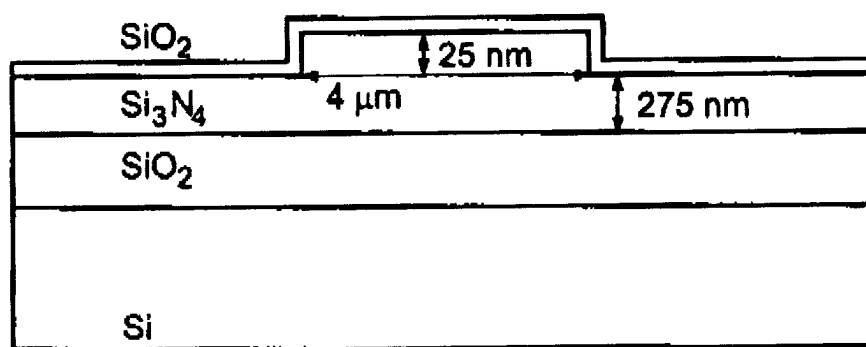
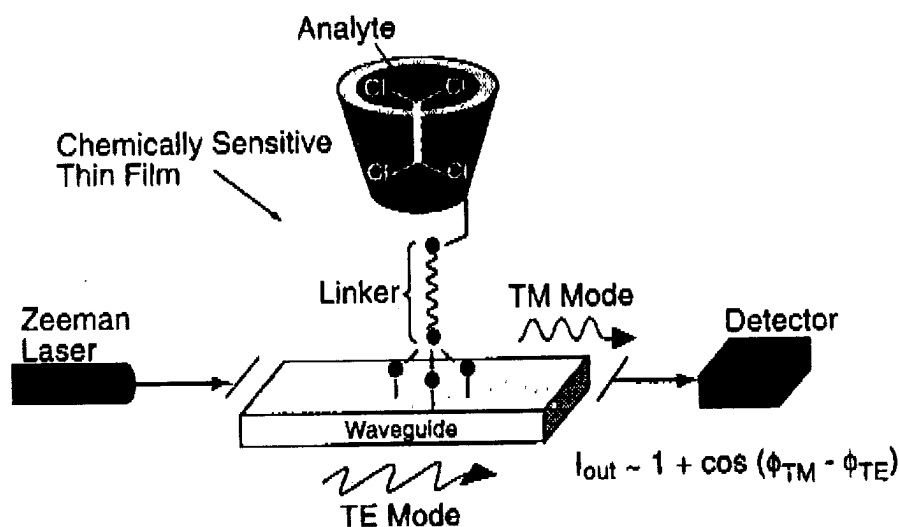


Figure 1. a) Schematics of the integrated optics thin-film sensor employing waveguide Zeeman interferometry, b) the cross section of the Si_3N_4 waveguide.

Fabrication

The waveguide cross section is depicted in Fig. 1b. Using a low pressure chemical vapor deposition (LPCVD) a 300 nm thick Si_3N_4 film was deposited on an oxidized Si wafer. Rib waveguides with different widths were formed by etching 25 nm high mesa structures into the Si_3N_4 film. A very thin top silica film was deposited using plasma enhanced chemical vapor deposition (PECVD), and finally the waveguide surface was coated with a monolayer film based on covalent attachment of heptakis-(2-0-methyl)- β -cyclodextrin to a C_{16} alkane linker layer [5].

Measurements

We measured the response of this thin-film integrated optics chemical sensor by exposing it to different concentrations of toluene in air. The measurements were performed with 2 cm long and 4 μm wide waveguide and using microscope objectives to couple light in and out of the waveguide. The relative phase change was measured using conventional lock-in detection techniques. Figure 2 shows the measured relative phase change when the device was exposed successively, for a duration of about 1 min, to three different toluene concentrations (200 ppm (parts per million), 100 ppm, and 50 ppm). The measurement clearly demonstrates the real-time ($\sim \text{sec}$) and reversible sensor response. The sensor also exhibits good linearity. In Fig. 3 the measured response under exposure to toluene at 20 ppm level is shown to demonstrate the device sensitivity in the low ppm range.

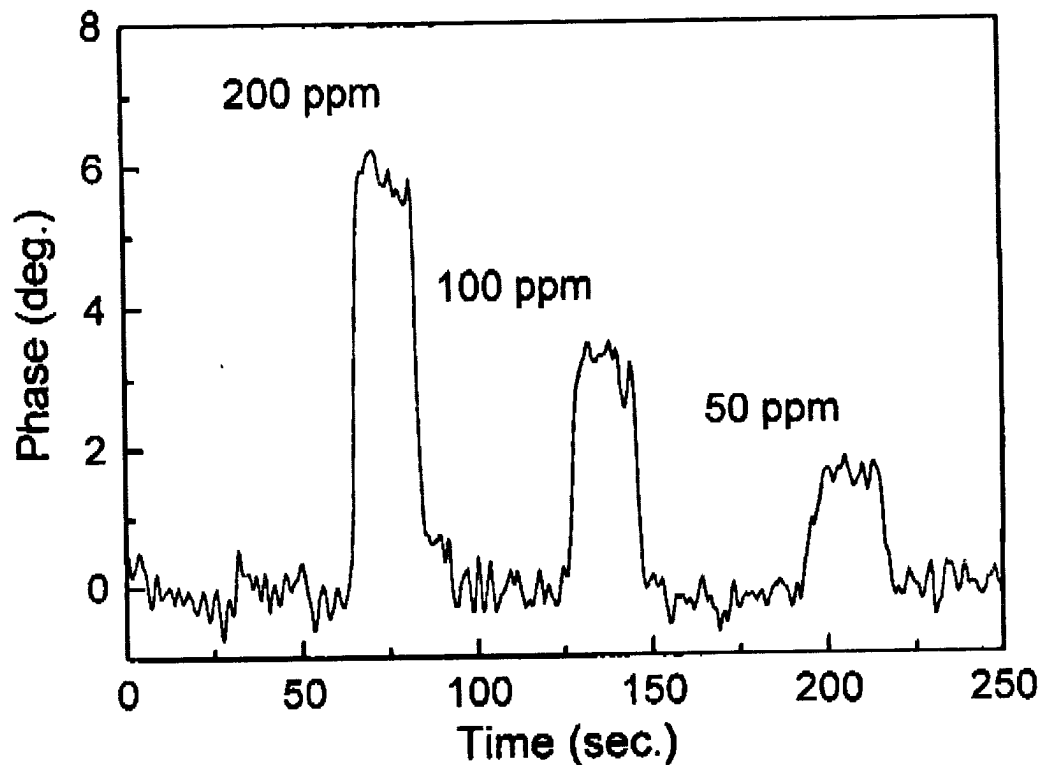


Figure 2. Measured sensor response under successive exposures to three different concentrations of toluene (200 ppm, 100 ppm, and 50 ppm).

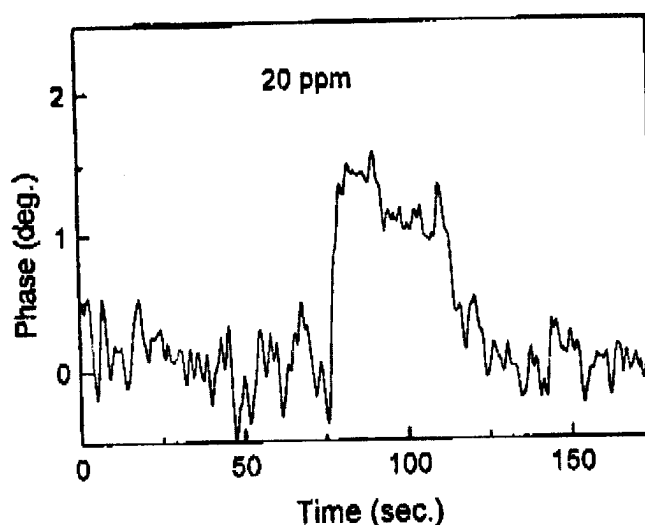


Figure 3. Measured sensor response under exposure to 20 ppm toluene in air.

Conclusion

We have demonstrated a new scheme for integrated optical chemical sensors utilizing Zeeman interferometry with Si_3N_4 waveguides coated with species-selective thin films. Our waveguide Zeeman interferometry is a simple, highly sensitive and stable detection method. The Si_3N_4 waveguide technology is extremely well established. The waveguide fabrication is fully compatible with microelectronics processing. Our results presented here demonstrate comparable sensitivities between the waveguide Zeeman interferometer and a conventional 250 MHz SAW transducer coated with the same film [3]. Furthermore, we believe that significant increases in sensitivity can be readily achieved through the optimization of the waveguide structure and the optical pathlength as well as the minimization of the system phase noise.

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References

1. G. Boisdé and A. Harmer, *Chemical and biochemical sensing with optical fibers and waveguides*, Artech House, Boston (1996).
2. W. Lukosz, "Integrated optical chemical and direct biochemical sensors", *Sensors and Actuators B* 29, (1995), pp. 37-50.
3. L.W. Moore, K. N. Springer, J-X. Shi, X. Yang, B.I. Swanson, and D. Li, "Surface acoustic wave chemical microsensors based on covalently bound self-assembled host monolayers", *Advanced Materials* 7, (1996), pp. 729-731.
4. R. G. Johnston and W. K. Grace, "Refractive index detector using Zeeman interferometry", *Appl. Opt.* 29, (1990), pp. 4720-4724.
5. B. Swanson, J. Shi, S. Johnson, and X. Yang, in proc. ACS Symposium on Chemistry and Technology of Chemical and Biosensors, Orlando, Florida, August 1996, in press.



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