

# Silicon Chip Based Near-Infrared and Mid-Infrared Optical Spectroscopy for Volatile Organic Compound Sensing

Yi Zou,<sup>1</sup> Swapnajit Chakravarty,<sup>2</sup> Xiaochuan Xu,<sup>2</sup> Weicheng Lai,<sup>1</sup> and Ray T. Chen<sup>1</sup>

<sup>1</sup>Microelectronics Research Center, Electrical and Computer Engineering Department, University of Texas at Austin, Austin, TX, 78758, USA

<sup>2</sup>Omega Optics Inc., Austin, TX, 78759, USA Author

e-mail address: yzou@utexas.edu, swapnajit.chakravarty@omegaoptics.com, raychen@uts.cc.utexas.edu

**Abstract:** We compared different on-chip silicon based absorption sensors for the detection of xylene in water in both near-infrared and mid-infrared with detection limit down to 1ppb. Strip waveguide, slot waveguide and PC-based chip integrated optical absorption spectroscopy devices are compared in near-infrared and mid-infrared. The results provide a route for enhanced sensitivity while retaining device miniaturization.

**OCIS codes:** (300.6340) Spectroscopy, Infrared; (230.7370) Waveguides

Infrared (IR) absorption spectroscopy is widely accepted as the ideal technique for chemical sensing due to the unique capability to distinguish analytes of interest based on unique molecular vibration signatures [1,2]. It has an overwhelming advantage over other methods that depend on sensing changes in refractive index. In our previous work, PC slot waveguide structures successfully detected and identified VOCs in liquid and gaseous phases for xylene in water and methane in nitrogen respectively [3, 4]. Results showed the enhancement due to slow light effect of photonic crystal waveguide (PCW) greatly reduces the total interaction length with chemical analytes. The length of PC slot waveguide for sensing xylene in water is only 300 $\mu\text{m}$ , which showed that the miniature PCW structure is an ideal platform for chip integrated optical absorption spectroscopy. Miniaturization enables the potential for multiplexed detection for the simultaneous identification and quantification of multiple organic compounds of interest. We also successfully detected xylene and trichloroethylene in water simultaneously by multiplexing PCW with different lattice constants [5]. In this paper, we compare absorbance of different waveguide devices in the near-infrared. We also show the working principle of our devices in the mid-infrared in silicon on sapphire (SOS) where stronger absorption peaks of the analytes of interest further improve our detection limit.

The principle of operation of optical absorption spectroscopy is governed by the Beer–Lambert law. According to this law, the transmitted intensity  $I$  is given by

$$I = I_0 \times \exp(-\gamma\alpha L) \quad (1)$$

where  $I_0$  is the incident intensity,  $\alpha$  is the absorption coefficient of the medium,  $L$  is the interaction length, and  $\gamma$  is the medium-specific absorption factor determined by dispersion-enhanced light–matter interaction. For various applications,  $L$  must be large to achieve high sensitivity since  $\gamma=1$ . In addition, from perturbation theory

$$\gamma \propto f \times \frac{c/n}{v_g} \quad (2)$$

where  $c$  is the speed of light in free space,  $v_g$  is the group velocity in the medium, and  $n$  is the refractive index of the medium [6]. The term  $f$  is the filling factor denoting the relative fraction of optical field residing in the analyte medium. Group velocity  $v_g$  is inversely proportional to the group index  $n_g$ . Hence, theoretically, the optical absorbance by a waveguide on a same chip increases in order as follows in silicon: (a) strip waveguides, ( $n_g \sim 3$ ), (b) slotted strip waveguides ( $n_g \sim 3$ ,  $f \sim 10$ ) since the intensity of light in a low-index slot is significantly enhanced compared to strip waveguides, (c) PCWs ( $n_g \sim 100$ ) [7], and (d) slotted PCWs ( $f \sim 10$  and  $n_g \sim 100$  for a combined factor of  $\sim 1000$ ). In mid-infrared, fundamental vibration signatures of organic compounds are stronger by two to three orders of magnitude in general than their corresponding overtones in the near-infrared. Hence, due to an increase in  $\alpha$  in Eq. 1, together with the device enhancements from Eq 2., mid-infrared spectroscopy can be expected to have a larger sensitivity in absorption spectroscopy than in the near-infrared.

For near-infrared, the devices are fabricated in silicon-on-insulator (SOI) substrate using standard e-beam lithography and reactive ion etching (RIE). Three types of photonic devices including single mode strip waveguide, slot waveguide and photonic crystal waveguide are fabricated simultaneously onto the same chip. After that, a 2  $\mu\text{m}$  thick SU-8 polymer is spun onto the chip as a microextractor for volatile organic compounds (VOCs). The single mode strip waveguide is 500nm wide as shown in Fig. 1(a), while slot waveguide, shown in Fig. 1(b), has 80nm wide slot and 280nm wide rails. W1 PCW with lattice constant  $a=405\text{nm}$  shown in Fig. 1(d) and bandedge near 1674nm is also used for xylene detection. It is estimated from simulations, that the group index of the guided mode at 1674 nm for the individual waveguides is 33 [8]. Fig. 2 show the experimentally determined absorbance of xylene

in water. The 2  $\mu\text{m}$  thick SU-8 ensures negligible overlap of the propagating optical mode with ambient water. The absorbance increases with time when a higher concentration is added. All measurements are done 10 min after sample addition, which is a typical time observed in measurements beyond which no further change in absorbance is observed. The absorbance is calculated by comparing the transmitted light output at each concentration to the transmitted light intensity with no VOC. The typical absorption spectrum when  $10^{-7}\%$  xylene in water was added for slot waveguide is shown in Fig. 3(a).

In mid-infrared, silicon on sapphire (SOS) substrate. The wavelength adopted is at 3.4  $\mu\text{m}$  which has a stronger absorption peak for xylene. Furthermore, PDMS (poly dimethyl siloxane) is used as a microextractor due to its weaker absorbance than SU-8 in this wavelength range. Devices shown in Fig. 1(d) are fabricated using ebeam and inductively coupled plasma (ICP) etcher. Light emitted from an Interband Cascaded Laser (ICL) is coupled into the SOS chip through subwavelength grating couplers and single mode mid-infrared optical fibers. After passing through single mode strip waveguide and output grating coupler, the light is collected by an InSb detector. A reference InSb detector measures the fluctuations in the source laser as a function of time. In order to improve the signal-to-noise ratio, a mechanical chopper is used with chopping frequency of  $\sim 1\text{KHz}$ , and the detected signals from InSb are demodulated by a lock-in amplifier. Time scanning of power change is plotted in Fig. 3 (b) when  $10^{-7}\%$  xylene in water is introduced. The absorbance of xylene for mid-infrared strip waveguide is also plotted in Fig. 2, after subtraction from the reference. The drop in the signal observed in Fig. 3(b) upon introduction of xylene in water has a component from the absorbance of water, due to the interaction between water and the longer tail from the mid-infrared propagating optical mode. The contribution from water is independently determined by dispensing a drop of pure water on the chip. The same volume of analyte is chosen for each measurement. The net change due to xylene in water, at each concentration, was calculated by subtracting from the drop caused by water only. The PDMS thickness at 3.4 $\mu\text{m}$  is 8 $\mu\text{m}$ . An optimized thickness, balancing the contribution from water and measurement time, is being investigated.

We plot the trend of absorbance of xylene in different devices. The optical absorbance of the microextracting polymer also plays an important role in minimum detectable sensitivities. PC slot waveguide devices in the near-infrared were previously demonstrated in PDMS. The lower sensitivity observed in PC slot waveguides in the near-infrared is due to the lower signal-to-noise ratio with PDMS. PC slot waveguide devices are currently being measured with SU8. Mid-infrared strip waveguide show high absorption sensitivity than all other devices. Higher sensitivity, lower than 1ppb observed with near-infrared PCWs, is expected for mid-infrared PCWs in SoS currently being investigated.

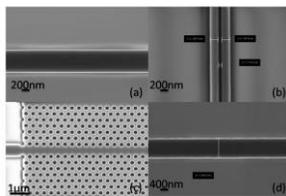


Fig. 1. SEM images of fabricated devices on SOI substrate (a) single mode strip waveguide, (b) slot waveguide, (c) W1 type PCW, and (d) single mode strip waveguide on SOS substrate.

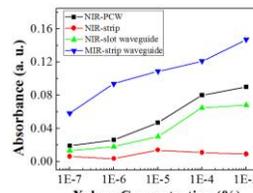


Fig. 2. Absorbance of xylene measured at 1674 nm with strip waveguide (red), slot waveguide (green), PCW (black) for NIR and measured at 3.4  $\mu\text{m}$  with strip waveguide (blue) for MIR.

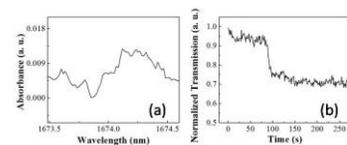


Fig. 3. (a) Absorbance of  $10^{-7}\%$  xylene in water for slot waveguide in NIR, (b) Time scanning of normalized transmission from strip waveguide in MIR before and after adding  $10^{-7}\%$  xylene in water.

## References

- [1] M. Lackner, "Tunable Diode Laser Absorption Spectroscopy (TDLAS) in the Process Industries-a Review," *Rev. Chem. Eng.* 23, 65 (2007).
- [2] F. Adler et. al., "Mid-infrared Fourier transform spectroscopy with a broadband frequency comb," *Opt. Express* 18, 21861 (2010).
- [3] W.-C. Lai, S. Chakravarty, X. Wang, C. Lin, and R. T. Chen, "Photonic crystal slot waveguide absorption spectrometer for on-chip near infrared spectroscopy of xylene in water," *Applied Physics Letters* 98, 023304 (2011).
- [4] W.-C. Lai, S. Chakravarty, X. Wang, C. Lin, and R. T. Chen, "On-chip methane sensing by near-IR absorption signatures in a photonic crystal slot waveguide," *Optics letters* 36, 984–986 (2011).
- [5] W.-C. Lai, S. Chakravarty, Y. Zou, and R. T. Chen, "Multiplexed detection of xylene and trichloroethylene in water by photonic crystal absorption spectroscopy," *Optics letters* 38, 3799–3802 (2013).
- [6] N. A. Mortensen and S. S. Xiao, "Slow-light enhancement of Beer-Lambert-Bouguer absorption," *Appl. Phys. Lett.* 90, 141108 (2007).
- [7] M. Notomi, K. Yamada, A. Shinya, J. Takahashi, C. Takahashi, and I. Yokohama, "Extremely Large Group-Velocity Dispersion of Line-Defect Waveguides in Photonic Crystal Slabs," *Phys. Rev. Lett.* 87, 253902 (2001)
- [8] C.-Y. Lin, X. Wang, S. Chakravarty, B. S. Lee, W.-C. Lai, and R. T. Chen, "Wideband group velocity independent coupling into slow light silicon photonic crystal waveguide," *Appl. Phys. Lett.* 97, 183302 (2010)

## Acknowledgements

The authors acknowledge the National Science Foundation (NSF) for supporting this work under SBIR program (IIP-1127251).