Integrated strip and slot waveguides in silicon-on-sapphire for Mid Infrared VOC detection in Water

Yi Zou,^{1,*} Harish Subbaraman,² Swapnajit Chakravarty,^{2,*} Xiaochuan Xu,² Amir Hosseini,² Wei-Cheng Lai,¹ and Ray T. Chen^{1, 2, *}

¹Dept. of Electrical and Computer Engineering, University of Texas, 10100 Burnet Road Bldg. 160,

Austin, TX, USA 78758;

²Omega Optics Inc., 8500 Shoal Creek Blvd., Austin, TX 78757

ABSTRACT

A chip integrated infrared spectrometer for in situ sensing and spectroscopic identification of VOCs in water probing large absorption cross sections of VOCs in the mid-infrared is desired. Preliminary strip and slot waveguide devices fabricated in silicon-on-sapphire for operation at $3.4\mu m$ wavelength experimentally demonstrated propagation loss of 2.1dB/cm for strip waveguides and 11dB/cm for slot waveguide. VOC are extracted from water using PDMS for solid phase micro-extraction and enables absorbance measurements independent from the strong absorbance of water. Absorbance of xylene as a typical VOC is determined on chip from the difference in transmitted intensity in the presence and absence of xylene.

Keywords: spectroscopy, mid-infrared, waveguides.

*yzou@utexas.edu, swapnajit.chakravarty@omegaoptics.com, raychen@uts.cc.utexas.edu; phone 1 512-471-4349; fax 1 512-471-8575;

1. INTRODUCTION

Infrared (IR) absorption spectroscopy has a unique capability to distinguish analytes of interest based on unique molecular vibration signatures, hence it is widely accepted as an ideal technique for chemical sensing [1,2]. It has an overwhelming advantage over other methods that depend on sensing changes in refractive index which is lacking of specificity to identify the exact material.

In our previous work, we successfully detected and identified VOCs in liquid and gaseous phases for xylene in water and methane in nitrogen respectively [3, 4] by using PC slot waveguide structures. Combining slow light effect of photonic crystal waveguide (PCW) and electric field intensity enhancement inside the slot structure the total interaction length with chemical analytes is greatly reduced to only $300\mu m$, which has the potential for chip integrated optical absorption spectroscopy. Benefiting from the device miniaturization we also successfully detected xylene and trichloroethylene in water simultaneously by multiplexing PCW with different lattice constants [5]. Since the large absorption cross-sections can be provided in the mid-infrared that increases the sensitivity to analyte detection, therefore it is expected if we can build on-chip absorption spectroscopy device in mid-infrared range we can get even better device performance.

As proposed by Soref et. al in Ref. 6 and 7, silicon is an ideal platform for mid-infrared applications up to 8 μ m benefiting from its low material losses, mature fabrication technologies, and high refractive index. Traditional silicon on insulator (SOI) wafer has been widely used in near-infrared, but it is limited by high absorption of silicon dioxide in the range between 2.6 to 2.9 μ m and above 3.5 μ m. Hence, silicon on sapphire (SOS) was proposed as an alternative platformwhich has transparent window up to 5.5 μ m, still providing a high refractive index contrast between core layer and the cladding [6, 7].

In this paper, we demonstrate strip waveguide and slot waveguide fabricated in silicon-on-sapphire (SOS) for transverse-electric (TE) polarized wave operation at $3.4\mu m$ wavelength. A propagation loss of 2.1dB/cm and 11dB/cm for strip waveguides and slot waveguides, respectively has been experimentally observed. We also demonstrated xylene sensing by using strip waveguide in mid-infrared.

Silicon Photonics IX, edited by Joel Kubby, Graham T. Reed, Proc. of SPIE Vol. 8990, 89900X © 2014 SPIE · CCC code: 0277-786X/14/\$18 · doi: 10.1117/12.2040412

2. DEVICE WORKING PRINCIPLE

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

$$I = I_0 \times \exp(-\gamma \alpha L) \tag{1}$$

where I_0 is the incident intensity, α is the absorption coefficient of the medium, L is the interaction length, and γ 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

$$\gamma \propto f \times \frac{c/n}{n_g} \tag{2}$$

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 [8]. 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 .

Mid-infrared covers most characteristic absorption bands of various organic and inorganic compounds that contain the molecular fingerprints, it has become very attractive for sensor applications. Compared with near-infrared spectroscopy, most of the chemical bonds have much stronger absorption peaks in mid-infrared than near-infrared, so by moving from near-infrared to mid-infrared the sensitivity of spectroscopy can be greatly enhanced by several orders.

At the same wavelength, since absorption coefficient α does not changed, the way to improve sensitivity while keeping the same device length L is to increase γ . As shown in Eq. 2, increase f or n_g or both of them two can lead to increase γ and hence enhance sensitivity. Slot waveguides have demonstrated in near-infrared that it can significantly increase the electric field intensity in the narrow slot which is filled with low index material. The enhancement can be around 10 times strong than conventional waveguide [9]. Photonic crystal waveguide has also been demonstrated in near-infrared that it can slow down the group velocity up to 100 [10]. These two devices provide the potential methods to increase γ and hence sensitivity. By considering these two factors theoretically we can expected strip waveguide, slot waveguide, photonic crystal waveguide and slotted photonic crystal waveguide on the same chip will have optical absorbance increases in order as follow: (a) strip waveguides which has $n_g \sim 3$, (b) slotted strip waveguides which has $n_g \sim 3$ and $f \sim 10$ since the intensity of light in a low-index slot is significantly enhanced compared to strip waveguides, (c) PCWs which has $n_g \sim 100$, and (d) slotted PCWs which has $f \sim 10$ and $n_g \sim 100$ for a combined factor of ~ 1000 .

Hence, due to an increase in α 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.

3. DEVICE DESIGN

All the devices are on SOS platform which has 600nm silicon layer on top of 500µm handle sapphire substrate. Fig. 1(a) shows the designed single mode strip waveguide working at 3.4µm. The device has 1µm width and 600nm height, hence it can only support fundamental mode for TE polarized wave inside the waveguide. The slot waveguide which has 600nm rail width, 130nm slot width and 600nm height on SOS platform is shown in Fig. 1(b). This configuration results a confinement factor of 0.39, as we can see in the figure, that a strong electrical field intensity is confined inside the narrow slot. The enhancement of electrical field intensity will further improve light-matter interaction, and hence lead to sensitivity improvement.



Fig. 1. Simulated electrical field of (a) strip waveguide, and (b) slot waveguide.

4. DEVICE FABRICATION

The devices are fabricated on a SOS platform with 600nm silicon device layer on a 500µm thick sapphire substrate. The fabrication flow is shown in Fig. 2. A 140nm silicon dioxide layer is first deposited on top of silicon layer using plasma enhanced chemical vapor deposition (PECVD) to serve as a hard mask for pattern transfer. All the components including grating couplers, strip waveguides, slot waveguides and strip-to-slot waveguide mode converters are patterned in one step with JEOL JBX-6000FS electron-beam lithography tool using ZEP-520A ebeam resist, followed by developing in n-Amyl acetate (ZEP-N50) for 2mins, and rinsing in isopropyl alcohol (IPA). The ebeam resist pattern is next transferred to silicon dioxide by reactive ion etching (RIE) using CHF3 and O2 at 400V DC bias and 40mTorr pressure for 8mins. Following this, the pattern in silicon dioxide is transferred to silicon by inductively coupled plasma (ICP) etch using HBr and Cl2at 400W ICP power, 200W RF power, 10mTorr pressure and 20Torr Helium flow for backside cooling for 6.5min. Finally, the chip is cleaned using Piranha for 15min and followed with three cycles of Piranha/HF post-process treatment as suggested from Ref. [11]. Similar to previous demonstration of PC slot waveguide in xylene detection, PDMS top cladding was prepared by spinning a 10:1 mixture of Sylgard Elastomer 184 from Dow Corning, NY and curing agent, followed by oven baking for 1 hrs at 90°C. PDMS is hydrophobic, hence the absorption spectrum of VOCs can be obtained independent of any interference from the strong absorbance signatures of water in mid-infrared.



Figure 2: Fabrication steps of strip waveguides and slot waveguide (a) PECVD growth of oxide (b) E-beam Resist (ZEP-520A) patterning (c) Transfer of resist pattern to oxide by RIE using CHF3 followed by resist strip (d) Transfer of pattern from oxide to Si by ICP in HBr and Cl2.

SEM images of the fabricated devices are shown in Figs.3 (a)-(b).



Fig. 3. SEM images of fabricated devices on SOS substrate (a) single mode strip waveguide, and (b) slot waveguide.

5. EXPERIMENTAL RESULTS

In mid-infrared, SOS substrate, the wavelength adopted is at 3.4μ 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. Light emitted from an Interband Cascaded Laser (ICL) is coupled into the SOS chip through 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 ~1KHz, and the detected signals from InSb are demodulated by a lock-in amplifier. Propagation losses of strip waveguides and slot waveguides are first characterized by using well known cut-back method. We achieve 2.1dB/cm propagation loss for single mode strip waveguide and 11dB/cm propagation loss for slot waveguide. Both of them are plotted in Fig. 4.



Fig. 4. (a) Measured loss of eight single mode waveguides fabricated on SOS operating at $3.4 \,\mu\text{m}$ wavelength. The waveguides are $0.6 \,\mu\text{m}$ in height and $1 \,\mu\text{m}$ in width. 2.1dB/cm propagation loss is achieved by linear fitting. (b) Measured loss of eight slot waveguide fabricated on SOS operating at $3.4 \,\mu\text{m}$ wavelength. A11dB/cm propagation loss is achieved by linear fitting.

Time scanning of power change is plotted in Fig. 5(a) when 10^{-7} % xylene in water is introduced. The absorbance of xylene for mid-infrared strip waveguide is plotted in Fig. 5(b), after subtraction from the reference. The drop in the signal observed in Fig. 5(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 3.4µm is 8µm. An optimized thickness, balancing the contribution from water and measurement time, is being investigated.

As a comparison, we also plot the absorbance we got from previous strip waveguide in near-infrared. As we can clearly see that the absorbance from mid-infrared device has much bigger absorbance than the device in near-infrared, as we expected, since the absorption coefficient of α in mid-infrared is much bigger than it in near-infrared. We also expected the slot waveguide with enhanced electrical field intensity will further improve the sensitivity. The xylene sensing experiment with mid-infrared slot waveguide is currently progressing.



Fig. 5. (a) Time scanning of normalized transmission from strip waveguide in MIR before and after adding 10^{-7} % xylene in water, and (b) Absorbance of xylene measured at 1674 nm with strip waveguide (black triangle) for near-infrared, and at 3.4 µm with strip waveguide (red square) for MIR.

6. SUMMARY

In summary, we demonstrate strip waveguide and slot waveguide fabricated in SOS for transverse-electric (TE) polarized wave operation at 3.4 μ m wavelength. Propagation loss of 2.1dB/cm has been experimentally observed for strip waveguides, while the propagation loss of slot waveguides is measured to be 11dB/cm. The absorbance of xylene in water is measured with strip waveguide in mid-infrared and compared with strip waveguide in near-infrared.

ACKNOWLEDGEMENTS

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

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] R. Soref, "Mid-infrared photonics in silicon and germanium," Nat. Photonics 4(8), 495 (2010).

[7] R. Soref, S. J. Emelett, and A. R. Buchwald, "Silicon waveguided components for the long-wave infrared region," J. Opt. A 8(10), 840 (2006).

[8] N. A. Mortensen and S. S. Xiao, "Slow-light enhancement of Beer-Lambert-Bouguer absorption," Appl. Phys. Lett. 90, 141108 (2007).

[9] Xu, Q., Almeida, V.R., Panepucci, R.R., and Lipson, M., "Experimental demonstration of guiding and confining light in nanometer-size low refractive index material," Optics Letters 29, 1626 (2004).

[10] Notomi, M. "Extremely large group-velocity dispersion of line-defect waveguides in photonic crystal slabs," physical Review Letters 87, 253902 (2001).

[11] R. Shankar, I. Bulu, and M. Loncar, "Integrated high-quality factor silicon-on-sapphire ring resonators for the mid-infrared," Appl. Phys.Lett.102(5), 051108 (2013).