Development of an integrated optical sensor on chalcogenide glasses and on porous silicon for the mid-infrared spectroscopy

(Student paper)

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This work presents the development of photonic integrated circuits based on chalcogenide glasses and porous silicon materials dedicated to mid-infrared (mid-IR) sensing. Optical losses of the different platforms are measured for a wavelength range spanning from 4.0 to 4.44 µm and 6.9 to 8.8 µm. At 4.28 µm, corresponding to carbon dioxide maximum absorption, propagation losses of 6.00 ± 0.46 dB/cm and 11.76 ± 0.2 dB/cm are demonstrated using, respectively, chalcogenide and porous silicon platforms. Based on these losses and interaction schemes between optical field and target molecules, either evanescent or volume detection, the expected sensitivity and limit of detection of both platforms are discussed. Experimental demonstration of $C0_2$ sensing in the mid-IR using both chalcogenide and porous silicon platforms is also provided.

Keywords: *Integrated Optics; Chalcogenide Glasses; Pourous Silicon; Optical Bench; Mid-Infrared; Sensing; Spectroscopy.*

1. INTRODUCTION

In recent years, there has been a growing scientific interest for the development of mid-infrared (mid-IR) photonic circuits dedicated to optical sensing applications [1] thanks to the remarkable progress in development of optical sources such as QCL and supercontinuum sources [2,3]. The presence, in this wavelength range, of the absorption bands displayed by several toxic and polluting molecules makes mid-IR sensor well placed to respond to a diverse range of users, in-situ and sometimes harsh environment applications. This level of performance has created an innovative need for materials and guiding structures. The use of integrated optical circuits as sensors has various advantages such as manufacturing cost and compactness of the packages. However, these circuits must be fabricated from mid-IR transparent materials. Chalcogenide glasses (ChG) and porous silicon (PSi) have a great potential for the design of such circuits. ChG glasses display extended transparency ranging from near infrared to 20 µm depending on their composition [4] whereas porous silicon is transparent from 1 to 8 µm [5]. ChG can be used for evanescent field spectroscopy sensing but interaction with targeted molecules can be further enhanced using PSi as the open pores enable volume detection. In this paper, we propose a suitable mid-IR optical bench setup developed to characterise the transmission of PSi ridge waveguides in the wavelength ranges of 4.0 - 4.44 µm and of ChG ridge waveguides in the wavelength ranges of 6.9 - 8.8 µm.

FABRICATION OF INTEGRATED OPTICAL CIRCUITS

Prior the fabrication step, the ridge waveguide structures were designed using a commercial software (FIMMWAVE, Photon Design) to obtain the geometrical dimensions, width (w) and height (h), ensuring single-mode propagation while maximizing, for evanescent wave detection, the evanescent power factor in the superstrate.

Platform based on Chalcogenide glasses

A 5 µm confinement layer of $Ge_{28.1}Sb_{6.3}Se_{65.6}$ was grown an n-type Si (001) substrate by RF magnetron sputtering. This layer was covered by a guiding layer of composition $Ge_{12.5}Sb_{25}Se_{62.5}$. Single mode ridge waveguides (w=6 µm) widths were then fabricated using a classical i-line photolithographic process followed by a dry etching procedure at low pressure combining reactive ion etching (RIE) and inductively coupled plasma (ICP) etching (5 sccm CHF_3, 5 mTorr, 75 W ICP, 25 W RF). The waveguides obtained at the end of the process had a guiding layer thickness of 1.9 µm and a width of 6 µm (Fig.1-a) with corresponding refractive index values are equal to 2.69 and 2.49 at 4.28 µm for the guiding and confinement layers respectively.

Platform based on Porous silicon

PSi guiding and confinement layers were prepared by electrochemical anodization of a heavily doped P (100) silicon substrate with a 5 mΩ.cm resistivity and using applied current densities of 50 and 100 mA/cm^2, respectively, for specific times. The electrolyte was formed by combining hydrofluoric acid (50 %) with ethanol and deionized water in the ratio of 2-2-1. Refractive index is tuned by changing the porosity that depends on applied current density. In this way, two layers were fabricated: a 2.3 µm guiding layer with a porosity of 60% and a 5µm confinement layer with a porosity of 73% with corresponding refractive indices equal to 1.83 and 1.47 at 4.28 µm respectively. As showed on Figure 1-b, single mode ridge waveguides of 6-µm width were then fabricated using photolithography and dry etching techniques [6].

A PDMS fluidic cell was used for the detection of CO_2 with different sensing interaction lengths for the two platforms (Figure1-c).

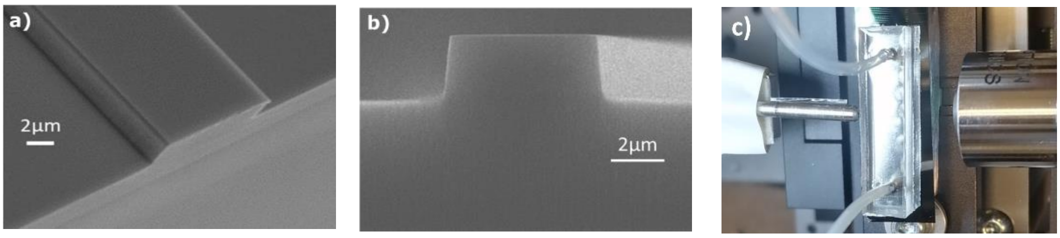

Fig. 1 . SEM Image of fabricated ridge waveguides (a) Chalcogenide glasses (b)Porous silicon. (c) Picture of the PDMS cell on a sample used for CO_2 sensing.

2. Optical bench and characterisation of guiding structures

Optical characterizations were performed by coupling the output of four tunable QCL emitting from λ=3.9 to 4.6 µm and from λ= 6.9 to 11 µm (MIRCAT from Daylight Solutions) into the ridge waveguides by using chalcogenide glass fiber. The output signal from the waveguide was collimated on a detector using a mid-IR objective lens. To improve signal to noise ratio, lock-in technique was used. The photolithography mask used consists of several sets of waveguides of different lengths. This configuration allows propagation loss measurements using a non-destructive cut-back method. Using mid-IR optical characterizations bench, PSi and ChG waveguides transmission was measured for wavelengths ranging from 4 µm to 4.44 µm. The same measurements were performed between

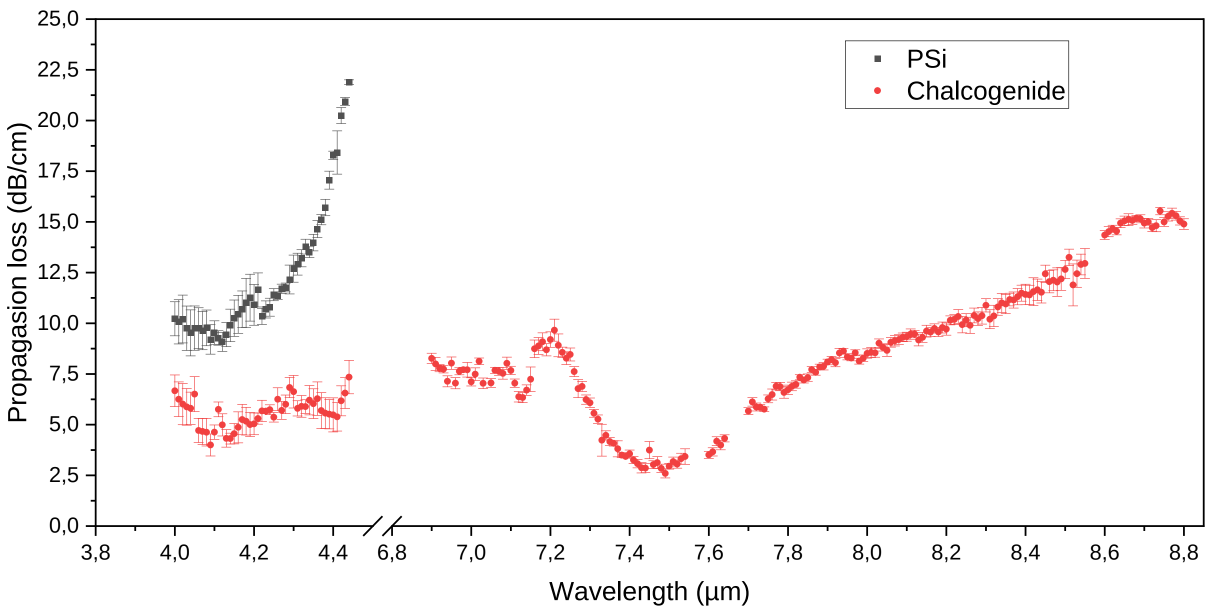

Fig. 2: Propagation losses as a function of the wavelength for porous silicon and chalcogenide glasses single mode waveguides. The laser does not cover the wavelength range between 4.6 and 6.9µm.

6.9 µm and 8.8 µm, only for ChG waveguides thanks to their extended materials transparency. To measure the propagation loss, a decreasing exponential function fit was applied to the measured signals for all waveguide lengths (from 8 to 13 mm with 0.5 mm step increment) and for each wavelength. The evolution of these losses in the two wavelength ranges [4.0 - 4.44 µm] for PSi and ChG waveguides and [6.9 - 8.8] µm for ChG waveguides is displayed in Figure 2. Higher propagation losses are expected from PSi due to volume diffusion but also due to the adsorption of different molecules present in ambient air inside the pores. The latter cause of losses are not expected in ChG, however additional propagation losses can results from the presence of a layer of a few nanometers of fluoropolymer [7] that is absorbent for this wavelength range. This layer is formed on the waveguide surface during the fluorine chemistry-based RIE etching.

Sensing demonstration

Transduction tests are carried out for CO_2 sensing. Figure 3-b shows the transduction results obtained from PSi waveguides. Using a PDMS cell (Figure 1-c) to make CO_2 – N_2 gas mixture in contact with the waveguide. CO_2 concentration up to 4970 ppm was detected around the two tabulated absorption lines of CO_2 around 4.23 µm and 4.28 µm [8]. The regeneration of the sensor application is demonstrated in figure 3-c by increasing and decreasing gradually the CO_2 concentration starting and finishing by a N_2 purge with a fixed laser wavelength emission at 4.26 µm.

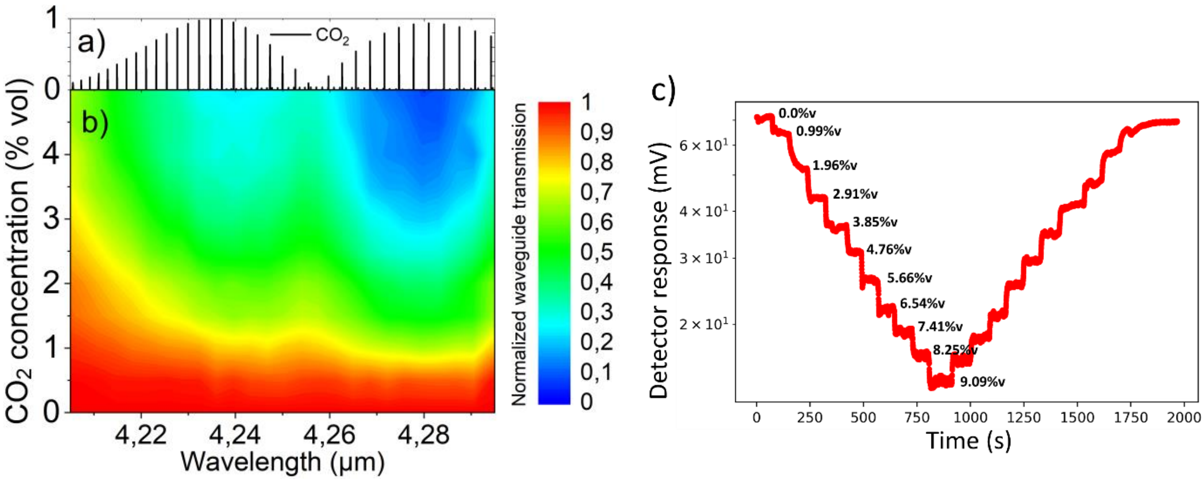

Fig. 3: (a) Normalized CO_2 absorption [8]. (b) Normalized waveguide transmission as a function of the wavelength for different CO_2 concentration. (c) Intensity evolution at λ=4.26µm for different CO_2 concentration.

Conclusion

The optical properties of chalcogenides glasses and porous silicon are promising for the development of an integrated optical platform for volume or evanescent wave sensing applications. Propagation losses are measured in the mid-IR from 4.0 to 4.44µm and from 6.9 to 8.8µm. Carbon dioxide sensing is demonstrated using porous silicon ridge waveguide on CO_2 absorption peak at 4.28µm. Technological processing optimization is actually performed for both sensing platforms to further reduce their respective limit of detection but also to achieve detection of higher CO2 concentrations using either evanescent wave and volume detection.

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