Optical interferometry on lateral porous silicon

Yingning He, Douglas Silva de Vasconcellos, Véronique Bardinal, David Bourrier, Éric Imbernon, Xavier Dollat, Thierry Leichle

OPTICAL INTERFEROMETRY ON LATERAL POROUS SILICON

Y. HE[1], D. SILVA DE VASCONCELLOS[1], V. BARDINAL[1], D. BOURRIER[1], E. IMBERNON[1], X. DOLLAT[1], AND T. LEICHLÉ[1*]

[1]LAAS-CNRS, Université de Toulouse, CNRS, Toulouse, France
**Corresponding author e-mail address: tleichle@laas.fr*

SUMMARY

We demonstrate the transducing ability of lateral porous silicon membranes (LPSi) using optical interferometry. To this aim, we use a Fourier Transform Infra-Red spectrometer (FTIR) coupled to a microscope stage equipped with an appropriate objective in order to overcome the difficulty to obtain interference signal from the LPSi membrane with small dimensions. We have recorded reflectance spectra upon filling the membrane with various solvents and the observed shifts of Fabry-Pérot fringe patterns indicate that the we are able to differentiate between solvents, thus providing a proof-of-concept of the LPSi interferometric transducer.

1. INTRODUCTION

In the last 20 years, there has been an increase interest in using porous silicon (PSi) elements in optical transduction schemes as a basis for label-free biosensing, taking advantage of the PSi large active area and convenient surface chemistry [1]. PSi optical biosensors are mostly relying on reflectance interferometry, where interference patterns are induced from light reflecting at the top and bottom of an adequately biofunctionalized PSi layer. The PSi layer is usually created by silicon anodization into a silicon wafer where the pores are oriented perpendicular to the wafer surface plane, resulting in vertical PSi. For real-time detection, PSi can be integrated into a microfluidic channel where the optical measurement is carried out through a fluidic cell [2].
We have recently proposed a unique fabrication process for the realization of porous silicon with lateral pores and the integration of porous membranes into planar microfluidic devices [3]. Vertical and lateral porous membranes integrated in planar microfluidics result in two distinct sensing configurations: the flow-over (FO) and flow-through (FT) configurations, as shown in Figure 1. While theoretical work has shown that with similar sensor footprints, FT sensor offers up to 20-fold improvement in response time [4], the experimental comparison between FO and FT sensing configurations was investigated with PSi microcavities and free-standing PSi membranes [5]. This study reveals some issues encountered with FO sensors where analytes or contaminants can be trapped within the closed-ended pores, leading to an overestimation of the sensor sensitivity.
The aim of this work is thus to study the possibility to carry out interferometric measurements with our fabricated LPi membrane since this configuration might lead to the future development of on-chip flow-through biosensors.

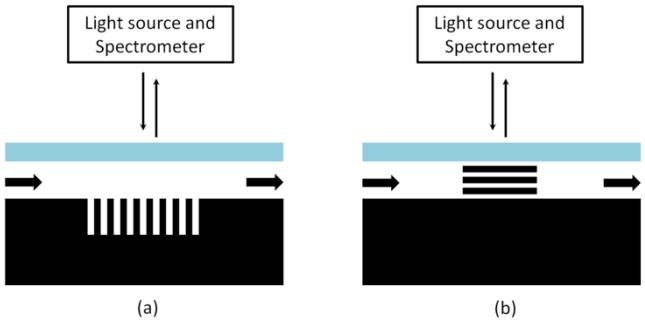

Figure 1: Two interferometric sensing configurations with PSi: (a) a typical flow-over configuration with a surface-based sensor within a microfluidic channel, (b) a flow-through configuration with an array of lateral nanopore tubes adequately connecting both microchannels.

2. EXPERIMENTAL RESULTS AND DISCUSSIONS

Measurements were carried out on previously fabricated lateral porous silicon chips [3], where each 1.6 cm×1.6 cm microfluidic chip bears two 5 µm deep microchannels separated by a 10 µm thick and ~4 µm deep porous silicon membrane with an average pore diameter of ~15 nm. Optical measurements were performed on a VERTEX 70 TIRF spectrometer connected to a HYPERION microscope stage (Figure 2). A 36× objective was used for localized measurements with the aim to collect a maximum signal from the micron scale porous silicon membranes. The porous silicon chip was loaded into a dedicated sample holder which was subsequently fixed on the microscope stage. The sample holder was

connected to a 4-channel reservoir for fluid management. The reservoir was connected to a pressure source from Fluigent to control the fluid flow inside the chip.
Various solvents were pumped into the microchannels and subsequently pushed through the LPSi membrane via positive pressure to ensure the full infiltration of the analyte into the membrane. Then, upon visual observation, the microscope was adjusted to focus on the top of the LPSi membrane and the observation window was downsized to a rectangular area of 10 µm × 100 µm using adjustable x- and y-apertures. These dimensions were chosen to fit the LPSi membrane (10 µm × 250 µm). Reflectance data acquisition was then carried out before flushing the analyte with a continuous air flow. To make sure that the analyte was completely removed, spectral measurements were taken during the flushing process until the spectrum was repositioned to that recorded for empty porous silicon. In order to test the interferometric transduction capability of the LPSi membranes, three kinds of solvents (water, ethanol, acetone) were tested.
Figure 3 presents the resulting reflectance spectra. Compared to empty and dried porous silicon, the presence of all three liquids clearly leads to a red shift of the spectrum corresponding to an increase in refractive index. The wavelength shifts from bare porous silicon are in the following order: water (n~1.328) < acetone ≈ ethanol (n~1.35), which is what expected, thus demonstrating the possibility to discriminate the various solvents.

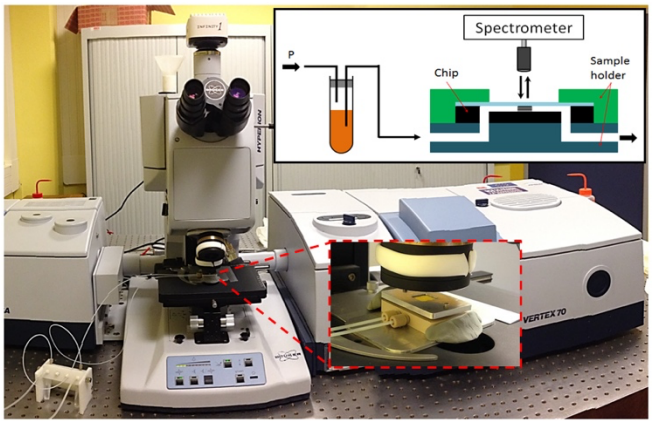

Figure 2: Experimental setup for microscale analysis and fluid-managing system used for optical detection.

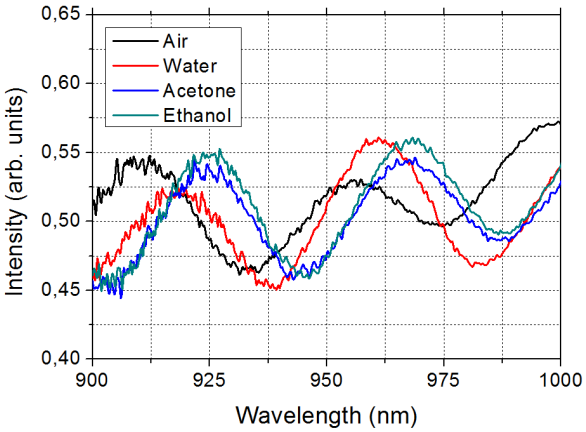

Figure 3: Reflectance spectra of a LPSi membrane filled with different solvents: water, acetone, and ethanol. The spectrum marked as air means that no liquid fills the pores.

3. CONCLUSIONS

In this work, we have demonstrated the Fabry-Pérot interferometric transduction capability of lateral porous silicon membranes bridging microfluidic channels. The specificity and difficulty of our study is that the LPSi membranes exhibit small dimensions, which has increased the difficulty to obtain clean and exploitable interference signals. Nevertheless, this issue was overcome by optimizing the experimental setup and the optical measurement conditions. We were able to identify the presence of solvents with different refractive index, thus providing a proof-of-concept of the LPSi interferometric transducer.

ACKNOWLEDGMENT

The authors acknowledge the Agence Nationale de la Recherche (ANR-17-CE09-0024-01). This work was partly supported by the French RENATECH network.

REFERENCES

[1] G. Shtenberg, E. Segal, *Handbook of Porous Silicon*, Springer International Publishing, **2014**.

[2] A. Janshoff, K. P. S. Dancil, C. Steinem, D. P. Greiner, V. S. Y. Lin, C. Gurtner, K. Motesharei, M. J. Sailor, R. Ghadiri, *Journal of the American Chemical Society*, **1998**, 120(46), 12108-12116.

[3] Y. He, T. Leïchlé, *Sensors and Actuators B: Chemical*, **2017**, 239, 628-634.

[4] C. Escobedo, A. G. Brolo, R. Gordon, D. Sinton, *Analytical Chemistry*, **2010**, 82(24), 10015-10020.

[5] N. Kumar, E. Froner, R. Guider, M. Scarpa, P. Bettotti, *Analyst*, **2014**, 139(6), 1345-1349.