Reflectance-based Photonic Crystal Liquid Sensors Made of ALD TiO$_2$

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Abstract

A promising concept for a photonic crystal sensor for liquid sensing applications is introduced. The two dimensional photonic crystals are fabricated using a recently developed Atomic layer deposition ARrays Defined by Etch-back technique (AARDE) to obtain large functional surfaces and dense pillar arrays. Photonic signals are collected by means of reflectance, which facilitates optical couplings and decreases fabrication steps. Large TiO$_2$ pillar arrays (3 mm × 3 mm in size) with pillar radius of 220 nm, height of 890 nm and pitch of 800 nm are realized. A 300 nm thick SiN membrane is used to seal the sensor and to prevent liquid evaporation, while allowing light penetration. To examine the sensing capability of the fabricated PCs, optical measurements are performed with liquids with various refractive indices (water, acetone and isopropanol). Explicit peaks are observed shifting towards higher wavelengths in the designed wavelength range of 1.35 – 1.55 µm (infrared) as the index of the measured liquid increases. A high sensitivity of 537 nm per refractive index unit is found for different peak series.

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Keywords: Photonic crystal, liquid sensor, reflectance, ALD TiO$_2$

1. Introduction

Photonic crystals (PCs) are synthetic structures, in which dielectrics change periodically, resulting in refractive index-sensitive photonic band gaps [1]. Therefore, photonic crystals can be used for various sensing applications [2]. Here, we propose a reflectance-based PC device as liquid sensor. The PC liquid sensor, schematically depicted in Fig.1a, consists of an ALD TiO$_2$ nanopillar array and a transparent
capping membrane which seals the sensing chamber. Polarized light beam is projected onto the cap window with an incident angle of \( \theta \), while the reflecting signals are symmetrically collected. Unlike the transmittance-based method, which requires complicated coupling steps and needs to take care of the loss issue, the reflectance-based method can result in easier packaging and coupling processes and larger functional surfaces, thus increasing the output signal.

![Schematic drawing of a PC liquid sensor](image1)

![SEM image (top view) of a fabricated TiO\(_2\) nanopillar array.](image2)

Fig. 1: (a) Schematic drawing of a PC liquid sensor; (b) SEM image (top view) of a fabricated TiO\(_2\) nanopillar array.

An ALD TiO\(_2\) PC sensor was recently reported to demonstrate the sensing ability by coating it with different PECVD oxides [3]. Results showed that the reflecting peak shifted to the higher wavelength range as the RI of the coated film increases. In this paper, preliminary coupling and packaging techniques are explored for the reflectance-based PC liquid sensors. Ultra thin SiN membranes are fabricated as cap windows. To verify the sensing capability, different liquids with similar RIs are optically tested with the same PC liquid sensor.

2. Device fabrication

Although TiO\(_2\) is a promising material with relatively high refractive index and high transparency in the visible and infrared regions, its hardness and chemical stability make it difficult to anisotropically dry etch into accurately dimensioned nanostructures. To obtain nanopillar arrays with high pillar density and large functional area, a so-called ALD ARrays Defined by Etch-back technique (AARDE) technique was employed to make two dimensional TiO\(_2\) PC structures [4]. In this paper, 3 mm \( \times \) 3 mm TiO\(_2\) pillar arrays with pillar radius of 220 nm, height of 890 nm and pitch of 800 nm, are realized using the AARDE technique (Fig. 1b).

In order to acquire low loss reflecting signals from the PC liquid sensors, smooth and transparent cap layers are needed to seal the functional chambers. Low-stress silicon nitride thin (300 nm) membranes are used as the cap layer. The large SiN windows (up to 1 cm \( \times \) 1 cm) are the formed by etching the silicon substrate in a KOH solution (Fig. 2a).

The sample for measurements is assembled as shown in Fig. 2b. Three different liquids, DI water, acetone and isopropanol, with similar refractive index of 1.333, 1.356 and 1.378, respectively, are used in sequence on the same device for the sensing tests. The liquid droplets are dropped onto the device area. The SiN cap window wafer is then placed on top of the device wafer and pressed on it. Surface tension helps to drive away the air and the pressing helps to seal the gaps. The reflectance measurements are performed with a spectroscopic ellipsometry (VASE, J. A. Woollam Co., Inc) at the incident angle of 60° using a transverse magnetic (TM) mode beam through the cap window along the \( \Gamma-M \) direction in the
photonic crystal arrays. After each measurement, the device and the cap wafer are rinsed and dried and re-used for the next test.

Fig. 2: (a) Optical image of a wafer containing the fabricated SiN cap windows and (b) a sketch of the assembling of the cap window on the PC liquid sensor structure.

3. Results and discussion

The measured reflectance signals of the three liquids are plotted in Fig. 3a. It is observed that only in a specific wavelength region, namely 1.35 – 1.55 μm in this case, clear response differences are detected. Two series of sharp peaks are found with higher reflectance values than in other regions. This suggests that a lower transmittance occurs, or that light is more difficult to propagate through the pillar arrays. This wavelength region is in good agreement with the simulated photonic band gaps of such TiO₂ PC structures (not shown here). According to the simulations, the band gap should be squeezed to smaller frequency if the environmental refractive index increases. This is confirmed by both series of peaks as listed in Table 1.

Fig. 3: The reflectance signals of different liquids (a); linear relationship between the wavelength of the sharp peaks and the liquid refractive index (b).
Table 1. Reflectance peak positions of different measured liquids

| Measured liquid | DI water | Acetone | Isopropanol |
|-----------------|---------|---------|-------------|
| Refractive index | 1.333   | 1.356   | 1.378       |
| Peak position: Series 1 (nm) | 1390.4 | 1406.0 | 1414.3 |
| Peak position: Series 2 (nm) | 1500.8 | 1513.0 | 1520.0 |

To study the sensitivity of the PC liquid sensor, the wavelengths of the peaks are plotted along the corresponding refractive index of the three liquids (Fig.3b). Linear relationships can be found for both of the peak series. A high sensitivity of 537.41 nm per refractive index unit (nm/RIU) is obtained for peak series 1. The second peak series shows a smaller sensitivity of 433.58 nm/RIU. Although the reason why there are two peak series is still unknown at the moment, such high sensitivity and the easier way of measuring compared to transmission, already makes the reflectance-based PC liquid sensors promising for various applications, such as real time quality monitoring in food and biomedical industry.

4. Conclusions

Reflectance-based photonic crystal liquid sensors with ALD TiO$_2$ nanopillar arrays are proposed. The 800 nm pitch uniform arrays are fabricated using the AARDE technique, resulting in both large functional areas and nanosize high pillars. Ultra thin SiN membranes with thickness of 300 nm and up to centimeters in size are used as cap windows for the reflectance measurements. Three different liquids with similar refractive index are tested with the same PC sensor. Results show that two sets of sharp reflectance peaks are observed only in a specific wavelength region. The peaks shift linearly towards higher wavelength as the refractive index increases. A high sensitivity of 537.41 nm/RIU is obtained. The high sensitivity and the relatively easier way of reflectance measurements as opposed to transmission, make the reflectance-based PC liquid sensors promising devices for various sensing applications.

Acknowledgements

The authors are thankful to the DIMES IC Processing group for their technical support. This work is supported by the TFN program of the Dutch Technology Foundation STW (Project 10026).

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