Photonic crystal slab sensor with enhanced surface area

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Abstract: In this work, we demonstrate improved molecular detection sensitivity for silicon slab photonic crystal cavities by introducing multiple-hole defects (MHDs), which increase the surface area available for label-free detection without degrading the quality factor. Compared to photonic crystals with L3 defects, adding MHDs into photonic crystal cavities enabled a 44% increase in detection sensitivity towards small refractive index perturbations due to surface monolayer attachment of a small aminosilane molecule. Also, photonic crystals with MHDs exhibited 18% higher detection sensitivity for bulk refractive index changes.

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OCIS codes: (230.5298) Photonic crystals; (230.5750) Resonators; (130.6010) Sensors.

References and links
Direct methods for sensing biomolecules could potentially eliminate the need for fluorescent labeling of molecules, which is both expensive and time-consuming [1]. Ideally, real-time detection would be performed using lab-on-chip platforms that require very small volumes of analyte. Silicon is attractive as a material platform for this type of monolithic, label-free biosensing due to its well-known processing techniques in the microelectronics industry, which could enable electrical integration and the ability to fabricate thousands of sensor devices on a single chip. Within the last decade, sensors utilizing silicon photonic devices such as resonant rings and disks [2–6], slot waveguides [7,8], and interferometers [9–11] have successfully achieved label-free detection of molecules such as proteins and DNA. Most commonly, detection is achieved by functionalizing the surface of a resonant cavity or waveguide, which can then be used to selectively capture a specific target molecule.

Recently, silicon photonic crystals (PhC) have also been used in refractive index sensing applications. The photonic bandgap that arises from these periodically patterned dielectric lattices can be utilized for relatively low-loss waveguiding [12,13] and microcavities with quality factors (Q) as high as $10^6$ [14–16], which are advantageous for monolithic sensor platforms. Moreover, PhC cavities could potentially have a smaller footprint compared to ring-based sensor devices while maintaining a high Q. PhC-based biosensors detect molecules attached to the surface (surface-based sensing) or liquids filling the volume that surrounds the PhC (bulk index sensing) via modulations of spectral features such as the waveguide cutoff [17,18] or microcavity resonance wavelength [19–21]. When analyte is exposed to a PhC sensor, the effective index of the waveguide or cavity mode is modified, causing a shift in the cutoff or resonance wavelength. The magnitude of the spectral shift is related to the spatial and temporal overlap of the mode with the analyte. In many PhC configurations, the spatial overlap is limited to the top surface of the defect region where molecules interact with an...
exponentially decaying field [22]. Thus, a key challenge for compact photonic crystal sensors is the limited available surface area for analyte binding.

In this work, we show that adding surface area to the defect region of PhCs, by directly placing holes with diameters much smaller than the lattice constant within silicon PhC slab microcavities (“multiple hole defects”, MHDs), improves the refractive index-change sensitivity. MHDs increase the number of available binding sites for molecules without compromising the Q of the cavity, and they accordingly provide increased spatial overlap of the resonant mode with molecules. We previously demonstrated through finite-difference time-domain (FDTD) simulations that PhCs with MHDs exhibit larger resonance shifts and, consequently, improved sensitivity for detecting analyte compared to PhCs with single hole defects (SHD) [23,24]. SHD-based sensors have previously been demonstrated in L1 defects, which consist of a single point defect [20]. Larger defects, such as the L3, provide a larger initial surface area for the placement of MHDs, and often have larger Q than L1 defects. Here, we present experimental demonstrations that show how the addition of MHDs in fabricated L3 slab PhC cavities increases detection sensitivity without degrading the cavity Q.

2. Device design and simulation

We performed three-dimensional FDTD simulations in order to examine the effect of adding MHDs to an L3 cavity. All dimensions are normalized to the PhC lattice constant of 410 nm (‘a’) for cavity resonances in the range of 1550-1600 nm. The silicon slab forming the PhC is surrounded entirely by air, with a slab thickness of 0.536 a (220 nm) and lattice hole diameter 0.512 a (210 nm). The MHD consists of three “defect holes” located within the cavity with diameters ranging from 50 to 80 nm, which are spaced laterally by 470 nm from center. The two lattice holes laterally adjacent to the cavity are moved outwards by 0.15 a (62 nm) to increase the quality factor by relaxing the boundary conditions on the cavity mode Fourier transform [14]. The cavities are surrounded by 7 lattice rows in the Γ-M direction, and 10 rows in the M-K direction. The simulation resolution is set to 24 (grid points every a/24, or 17 nm) with subpixel averaging enabled to increase accuracy [25], and the entire simulation space is surrounded by a perfectly matched layer (PML) of thickness 2.0 a, absorbing at the boundaries any fields emitted by the cavity [26].

The resonance wavelength and quality factor of the cavities are calculated by placing a TE-polarized dipole point source with a Gaussian frequency distribution within the cavity, offset by 0.1 a (41 nm) in the X and Y directions in order to avoid direct excitation of field maxima/minima. The broadband pulse excites the cavity modes, and the highest Q resonance remains after many periods have been calculated. The resonance wavelength and quality factor are then computed by harmonic inversion software (harminv) [27], which decouples the cavity fields into individual sinusoids and calculates their decay rates.

The resulting cavity modes are shown in Fig. 1. The calculated resonance frequencies for the L3 cavity and MHD were 0.2626 c/a and 0.265 c/a respectively, corresponding to 1561 nm and 1547 nm for a lattice constant of 410 nm. The addition of defect holes filled with air lowers the modal index and blue-shifts the resonance. Examination of Fig. 1(a-b) shows similar in-plane mode profiles for both cavities, with three main lobes inside the cavity region. Figure 1(c-d) shows the plotted field amplitude for each cavity using a vertical cut through the slab center, with the matching field profile plot overlaid for reference. It is important to note that the field amplitude is continuous in the MHD, with the highest positive field directly overlapping with the central defect hole and lowest negative field at the lateral defect holes. This sharp field increase inside the defect holes results in a perceived offset in field intensity between the MHD and L3 cavities after normalization to the color map scale. However, the field amplitude in the MHD cavity is in fact comparable to that in the L3 cavity. The calculated intrinsic cavity Q values are ~27,000 for the L3 cavity, and ~24,000 for the MHD cavity. We note that these Q values are in good agreement with those reported for similarly designed L3 cavities [14]. While higher intrinsic Q can be achieved through additional design
optimization [15], ultra-high Q factors can introduce increased sensitivity to environmental changes, which reduces the robustness of the sensor performance. The slight degradation of Q after insertion of the three MHD holes is likely caused by scattering occurring at the silicon/defect hole interfaces. Since the field is most strongly concentrated in the regions of additional surface area, inside the defect holes, analyte that binds to the silicon surface can be detected directly inside the volume where the modal field intensity is highest.

Fig. 1. Cavity mode TE field profiles from FDTD analysis overlaid onto the in-plane dielectric structure for (a) L3 and (b) 3-hole MHD cavities, with their respective vertical cuts and overlaid field amplitude data shown in (c,d).

3. Fabrication and methods

PhC lattices were patterned on 200 mm SOI wafers (SOITEC) with 220 nm silicon device layer and 2 μm thick underlying buried oxide (BOX). The device layer thickness is chosen to suppress TM propagation in the 500 nm wide strip waveguides [28], and the BOX thickness is sufficient to optically isolate the guided modes from the substrate. All fabrication steps were carried out using the standard 200 mm CMOS line at IBM T.J. Watson Research Center. First, 50 nm of silicon dioxide was deposited via LPCVD to act as a hard mask for etching. The device layer was then patterned using a Leica VB6-HR 100 kV electron beam lithography (EBL) system using proximity correction to simultaneously yield the input/output strip waveguides, the bulk photonic crystal holes, and the MHD. Each device consists of a PhC cavity region as shown in Fig. 2(a) with 2 mm long waveguides bringing the input/output signal to the ends of the chip, and 75 nm-width nanotapers at the waveguide ends. Polymer spot size converters for mode matching to tapered fibers were fabricated on top of the silicon nanotapers by optical lithography [29]. Finally, a second optical lithography step was performed to mask all regions except for the PhC lattices; subsequent exposure to a buffered oxide etch released the devices from the substrate, creating an air-bridge structure.

Over 100 devices were fabricated on each 4.6 mm-wide chip, consisting of solid L3 cavities, single-hole defect (SHD) cavities, and MHD cavities as shown in Fig. 2(b-d), respectively. Three different base doses were used in the EBL step, resulting in PhC lattice hole diameters of 210 ± 10 nm. Each chip was cleaved through the polymer couplers and mounted on a XY stage for positioning. Polarization-maintaining tapered fibers (OZ Optics Ltd.) with spot size 2.5 ± 0.3 μm were mounted onto XYZ stages with piezo control for bringing the input/output signal to the chip. A broadband LED source (Agilent 83437A) was used as the input source, with the output spectrum measured by an optical spectrum analyzer...
(Agilent 86140B). All measured traces were normalized to the transmission of an on-chip strip waveguide. For cavity measurements, the wavelength sweep on the OSA was limited to the region around the resonance to maximize resolution.

![Fig. 2. (a) Fabricated PhC with L3 cavity showing coupling to input waveguide. The cavity configurations examined in this work are the (b) solid L3 cavity, (c) SHD, and (d) MHD. Scale bar indicates lattice constant, 410 nm.](image)

### 4. Cavity characterization

We first examine the cavity resonances, measuring the transmission spectrum in air ambient at room temperature. Figure 3(a) shows the measured resonances for a solid L3 cavity, as well as for SHD and MHD cavities with defect hole diameters of 57 nm and 54 nm, respectively. The addition of a single defect hole (SHD) blue-shifts the L3 resonance by 6.46 nm; the MHD cavity resonance is blue-shifted by 11.94 nm compared to the L3 resonance. Defect hole diameters were measured by SEM metrology, and are assumed to be accurate within ± 5 nm. Based on multiple measurements and Lorentzian curve fitting, we estimate the precision of the cavity resonance wavelength measurements to be ± 0.003 nm. Lorentzian curve fits were also used to extract the loaded $Q$ ($Q_L$) of each resonance. We found a similar $Q_L$ of 6500 ± 200 for the different cavity configurations.

A summary of the resonance shifts for SHD and MHD cavities with different defect hole diameters is shown in Fig. 3(b). As expected, the magnitude of the resonance shifts is directly proportional to the defect hole diameter. When the defect hole diameter is increased, the effective index of the cavity mode accordingly decreases, which leads to the resonance wavelength blue-shift. FDTD simulations were performed on cavities with similar defect hole diameters, and this data is also shown in Fig. 3(b). The experimental and calculated data have good agreement overall. The error between experiment and calculation for the smallest MHD cavity is probably due to the difficulty in etching high aspect ratio holes of this size with vertical sidewalls as well as increased sidewall roughness. We note that the blue-shift of the resonance after addition of defect holes and the similarity of the $Q_L$ among all three cavity configurations is expected from our simulation results in section 2. Hence, we have verified that high quality factor can be maintained after the addition of small holes within the defect region of PhCs, which opens the door to improved molecular detection using the increased surface area available for analytic interaction.
Fig. 3. (Color online) (a) Transmission spectra comparing the resonance of a L3 cavity (solid) to those of SHD (dotted) and MHD (dashed) cavities with similar defect hole diameter.
(b) Resonance shift relative to L3 cavity for SHD and MHD cavities as a function of defect hole diameter. Measured results are shown as solid squares/circles (SHD/MHD), data from FDTD simulations are shown in open squares/circles, and a linear fit (dashed line) is provided as a guide to the eye.

5. Sensing experiments

5.1 Bulk index sensing

In order to first benchmark the refractive index sensitivity of SHD and MHD cavities compared to L3 cavities, we exposed the different PhCs to a solution of known refractive index. A pipette was used to wet the entire surface of the chips with a commercially available silicone oil (n = 1.397, Gelest, Inc.). The refractive index of the medium surrounding the PhC slab was therefore increased by 0.397. We then measured the devices, normalizing to the transmission spectra of strip waveguides that were also covered with silicone oil. Table 1 shows the resonance wavelengths for L3, SHD, and MHD cavities before and after the addition of silicone oil. The “large signal” resonance shifts observed for the L3 cavities can be attributed to the index change of the PhC lattice holes and the medium surrounding the PhC slab. Hence, the large signal shift results from the shift of the entire PhC band structure. The SHD and MHD cavities experience an additional “small signal” shift due to the index change in the SHD and MHD defect holes. Hence, for the PhC with SHD and MHD cavities, the resonance shifts due to silicone oil can be attributed to both the shift of the band structure and the additional shift of the resonant state within the band gap.

Table 1. Resonance data for SHD and MHD cavities before and after wetting with silicone oil. (All units in nm)

<table>
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<th>Diameter</th>
<th>Air</th>
<th>Silicone Oil</th>
<th>Shift</th>
<th>Diameter</th>
<th>Air</th>
<th>Silicone Oil</th>
<th>Shift</th>
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<td>(L3)</td>
<td>1551.3</td>
<td>1584.2</td>
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<tr>
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<tr>
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<td>80</td>
<td>1523.8</td>
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</table>

From the data shown in Table 1, we find that the large signal shift is 32.75 ± 0.15 nm for two different L3 cavities. With the addition of SHDs and MHDs, the small signal shift provides up to an additional 18% increase in the resonance shift for the 80 nm MHD, compared to an 11% increase for the 84 nm SHD. We calculate the detection sensitivity of these MHD and SHD cavities to be 98 nm/RIU and 91 nm/RIU, respectively, compared to the L3 cavities that have detection sensitivity near 82 nm/RIU. These sensitivities are comparable.
to previously published work on bulk index sensing of L3 and single point defect PhC cavities [21]. In general, as the accessible volume for sensing is increased, especially in regions of high field concentration, the detection sensitivity of the PhCs improves. Hence, a single large hole could potentially hold the same bulk index detection sensitivity as multiple smaller holes.

5.2 Surface-based sensing

For surface-based sensing when molecules are attached only on the surface of the sensor, the MHD cavities provide substantially increased available sensing surface area. The ideal surface-based sensor would consist of many small defect holes that maximize the accessible surface area for molecular binding within the mode volume. In order to probe the surface-based sensing efficacy of the MHD cavities, we exposed the MHD PhCs to a solution containing small molecules. This solution contained 4% 3-aminopropyltriethoxysilane (3-APTES) in water and methanol. The 3-APTES molecule is often used to functionalize oxidized silicon surfaces as a linker molecule to organic molecules, and it forms as a 0.8 nm thick monolayer on the silica surface [30]. Due to the presence of the heat-sensitive polymer couplers, we chose to utilize the native oxide on the silicon surface instead of growing a more typical thermal oxide. Each PhC sample was exposed to 4% 3-APTES solution in a humid environment for 20 min., and then rinsed with DI water and baked at 100°C for 10 min. to evaporate any remaining solvent.

The transmission spectra of the devices were measured before and after 3-APTES attachment, and the resonance shifts were evaluated to determine surface-based detection sensitivity, as shown in Fig. 4. In the figure, the resonances in air are centered on the x-axis, which has the same scale for all three devices, in order to simplify comparison. The measured spectra were fit with Lorentzian curves to extract the resonance wavelength and the QL. The resonances for the solid L3 cavity, which once again reveal the “large signal” contribution of the PhC lattice, and two sizes of MHD are included for comparison. The maximum surface-based sensitivity improvement compared to the L3 is 44% for the 67 nm diameter MHD. The MHD cavity with smaller defect hole diameter (56 nm) gives a 27% sensitivity increase. Both MHD cavities have a QL of ~6000. We note that due to the imperfect coverage of the native oxide, we do not believe that a perfect monolayer of 3-APTES molecules formed in the cavities. Further process optimization is necessary for robust sensor performance.

From the results shown in Fig. 4, it is clear that the additional surface area provided by the MHD enables improved surface-based sensing sensitivity without degrading the cavity QL. The relative sensitivity improvement gained by adding MHD to an L3 cavity is greater for surface-based sensing than for bulk index sensing. This result is expected because the additional internal surface area provided by the MHD represents a larger fractional increase in the overall available surface area for molecular binding in the PhC compared to the fractional increase in volume provided by the MHD for bulk index sensing.
6. Conclusion

We have demonstrated the design and fabrication of multiple-hole defects in slab PhCs. SHDs and MHDs consisting of defect holes with diameters of 50-80 nm placed directly inside of an L3 cavity increased the overlap of the cavity mode and analyte, and enabled enhanced detection capabilities without compromising the cavity Q. Experiments carried out using a bulk refractive index change surrounding the PhC slab showed that the inclusion of MHDs increases the resonance shift by up to 18% compared to traditional L3 cavities. The experimental sensitivity for this bulk index change, which is on the order of 100 nm/RIU, is comparable to values reported for similar PhC-based sensors. The best performance of the MHD cavities was illustrated by binding a monolayer of 3-APTES molecules to the surface of PhCs with either solid L3 or MHD cavities. We demonstrated up to 44% improvement in surface-based detection sensitivity for the cavities incorporating MHDs. Our approach of adding small holes in regions of high field concentration may also increase the detection sensitivity of other silicon photonic refractive index sensors, including ring resonators and interferometers.