High Sensitivity Sensor Based on Porous Silicon Waveguide

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ABSTRACT

Porous silicon (PSi) waveguides are fabricated as a new platform for high sensitivity biosensors. Biomolecules infiltrated into the PSi waveguide increase the effective refractive index of the waveguide and change the angle at which incident light couples into a waveguide mode. Due to the high surface area to volume ratio of PSi and the confinement of optical energy in the region where the biomolecules reside, the waveguide resonance is very sensitive to small concentrations of infiltrated molecular species. A resonance width below 0.1° has been obtained, which is sufficient to detect one monolayer of DNA covering the pore walls. In this work, a prism is used for the waveguide coupling in an arrangement that is similar to traditional surface plasmon resonance (SPR) sensing. Theoretical analysis suggests that an optimized PSi waveguide resonant sensor will show a 60-fold improvement in sensitivity when compared to a conventional SPR sensor due to the enhanced interaction between the electromagnetic field and biological material.

INTRODUCTION

The need to detect chemical and biological species at very low concentrations has stimulated efforts to devise highly sensitive devices. Optical biosensors that exploit surface plasmon resonance [1], fluorescence [2], and reflection [3], have been used widely to analyze biomolecular concentrations and interactions. The large surface area to volume ratio (~100 m²/cm³) [4] of porous silicon (PSi) makes it a good candidate material for label-free biomolecular sensing. Different size target molecules can be accommodated by controlling the pore size from a few nanometers to several microns [4]. Measuring the reflectance shift of Fabry-Perot fringes from a single PSi layer has been demonstrated for DNA and protein detection [5]. However, since a thin film is not a resonant structure, the sensitivity of the single layer PSi biosensor is limited as there is no sharp feature in the optical spectrum for which to accurately monitor small reflectance shifts. Multilayer PSi microcavities have also been used to detect various biomolecules [6] [7]. The drawback of these devices is their several micron thickness, which increases the infiltration time. Moreover, since the largest magnitude change in the optical spectrum is due to a refractive index change in the central layer of the resonant cavity, a larger volume of biological material is needed to ensure sufficient infiltration and binding.

Recently, a new sensor structure has been proposed, where a resonant PSi waveguide consisting of two PSi thin film layers is constructed and biomolecules are infiltrated into the porous waveguide [8]. The detection mechanism is a resonance angle shift that occurs when infiltrated biomolecules cause an effective refractive index change of the PSi waveguide. In addition to being a thin resonant structure, the PSi waveguide sensor confines optical energy in the layer where the biomolecules reside. This leads to a substantial increase in sensitivity compared to
SPR sensors and other waveguide sensors for which the electric field is attenuating as it penetrates the biomolecules [9]. Rigorous calculations based on a pole expansion indicate that the resonant PSi sensor theoretically has a 60-fold improvement in sensitivity over conventional SPR technology [8].

SENSOR STRUCTURE

The PSi waveguide structure is shown in Figure 1. Following the Bruggeman effective medium approximation that links porosity to refractive index [10], the top PSi layer has lower porosity and thus higher index, and the lower PSi layer has higher porosity and thus lower index. Above the top layer is an air gap, which has lower refractive index than the PSi film. Therefore, as shown in Figure 1, total internal reflection at both interfaces of the top PSi layer, or waveguide layer, is possible. The propagating wave is excited by a beam incident on the prism base of incidence angle $\alpha$. An SEM image of an example waveguide is shown in Figure 2. The fabrication details are provided in the next section.

![Fig. 1. Resonant waveguide structure. Light is confined by total internal reflection in the low porosity (high refractive index) porous silicon layer with thickness, $d$. Confinement occurs due to the air gap above and the lower refractive index (high porosity) porous silicon layer with thickness, $D$, below.](image1)

![Fig. 2. SEM of porous silicon waveguide structure. The bright region on top is the low porosity waveguide layer and the darker region below is the high porosity porous silicon layer.](image2)

In order to couple light into the waveguide, a prism is placed in close proximity to the top surface of the waveguide layer. The air gap between the prism and top PSi layer surface is typically 1/8 - 1/4 of the incident beam wavelength. Due to total internal reflection at the prism base, an evanescent wave with the proper wave vector can couple into a waveguide mode if the light is incident at the proper angle.

There are specific design requirements for the thickness of the three layers that confine or guide light in the waveguide. First, since light is coupled into the waveguide from the top surface, the thickness of the high porosity layer, $D$, beneath the waveguide layer does not need to be optimized. It only needs to be sufficiently thick to prevent light leakage into silicon substrate. Secondly, the air gap thickness needs to be optimized. If the air gap is too thick, the coupling is not efficient and the resonance dip is shallow. A shallow resonance may disappear completely during subsequent functionalization steps necessary for the biosensor preparation that increase
the effective refractive index of the waveguide. If the air gap is too thin, over-coupling may occur. In this case, the air gap is so small that some optical energy in the waveguide back-couples into the prism via an evanescent wave and reaches the detector. This effectively increases the resonance width, which is not desirable for achieving high sensitivity biosensors. During measurements, real time adjustment of the air gap size is possible by adjusting the coupling pressure between the prism and the waveguide. Appropriate pressure is obtained based on the tradeoff between the resonance width and resonance depth. It is important to note that experimentally varying the coupling pressure only changes the resonance width and depth but has no obvious effect on the absolute position of the resonance angle.

The thickness of the waveguide layer also needs to be optimized to achieve optimal optical mode confinement. If the waveguide layer is too thick, there is not as much spatial confinement of optical energy so the interaction between light and target materials in the PSi waveguide is not maximized. If the waveguide layer is too thin, the waveguide mode will not be well-confined and will leak out of the waveguide or the waveguide mode may not exist at all. The optimal PSi waveguide thickness is calculated using the theory presented in [8] and then experimentally optimized. Figure 3 shows the waveguide resonance width (full width half maximum) for different waveguide layer thicknesses, using incident light at wavelengths of 633nm and 1550nm, respectively. For all samples, the high porosity PSi layer thickness, D, is greater than 1 micron, which is sufficient to prevent light leakage into the substrate. As observed from the figures, the waveguide resonance obtained using infrared (IR) light is about one order of magnitude narrower than the waveguide resonance obtained using visible light. This is expected due to absorption losses in the silicon.

![Graphs showing waveguide resonance width for different layer thicknesses](image)

**Fig. 3.** Optimization of waveguide layer thickness to achieve narrow waveguide resonance using (a) 633nm and (b) 1550nm light. The PSi is as-anodized.

**FABRICATION AND EXPERIMENTAL RESULTS**

The waveguide is formed by electrochemically etching p⁺ (0.01Ω·cm) silicon wafers in an electrolyte of 15% ethanoic hydrofluoric acid. By applying different current densities during etching, it is possible to control the porosity and etching rate and create distinct PSi layers. Given the etching rate at a particular current density, the layer thickness is controlled by the etching duration. The two PSi layers are etched successively. The first layer is etched, then the applied current is changed and the second layer is etched in the same electrolyte.

For the narrowest as-anodized PSi waveguide resonance using 1550nm light, the etching specifications are given in Table I. The waveguide resonance based on these etching conditions is shown in Figure 4(a). The resonance width is about 0.1°. The waveguide is then oxidized at 900°C for 10 minutes for two purposes: to lower the waveguide loss [11], which also reduces the
resonance width [12], and to prepare the surface for subsequent surface chemistry functionalization procedures. After oxidation, the resonance width only reduces slightly, as shown in Figure 4(b), since PSi already has low loss in the IR range.

Table I: Etching condition for best IR resonance

<table>
<thead>
<tr>
<th>Layer</th>
<th>Porosity</th>
<th>Current Density</th>
<th>Thickness</th>
<th>Etching Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Waveguide</td>
<td>52%</td>
<td>5mA/cm²</td>
<td>300nm</td>
<td>60sec</td>
</tr>
<tr>
<td>Coupling</td>
<td>76%</td>
<td>48mA/cm²</td>
<td>1333nm</td>
<td>53sec</td>
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</table>

Fig. 4. Optimized resonance using 1550nm light before (a) and after (b) oxidation.

For the narrowest as-anodized PSi waveguide resonance using 633nm light, the etching conditions are given in Table II. The waveguide resonance from these etching conditions is shown in Figure 5. Before oxidation, the resonance width is about 0.7˚. After oxidation, the resonance is about 0.1˚. The resonance becomes narrower because oxidation reduces silicon absorption losses. Further oxidation reduces the losses even more; however, a mode may no longer be supported in the waveguide. Oxidation causes an effective refractive index decrease for both PSi layers and, in turn, decreases the refractive index contrast between the two layers. Having a smaller refractive index contrast requires a thicker waveguide layer to support waveguide modes. Oxidation, in fact, causes shrinkage of the low porosity waveguide layer [13]. In the case of the visible waveguide, the guiding mode is not supported after oxidation at 950˚ C.

Table II: Etching condition for best visible resonance

<table>
<thead>
<tr>
<th>Layer</th>
<th>Porosity</th>
<th>Current Density</th>
<th>Thickness</th>
<th>Etching Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Waveguide</td>
<td>52%</td>
<td>5mA/cm²</td>
<td>80nm</td>
<td>16sec</td>
</tr>
<tr>
<td>Coupling</td>
<td>76%</td>
<td>48mA/cm²</td>
<td>1235nm</td>
<td>49sec</td>
</tr>
</tbody>
</table>

Fig. 5. Optimized resonance using 633nm light before (a) and after (b) oxidation. Notice the different x-axis scales given for the two graphs.
**Water infiltration**

As a proof of principle experiment, water (refractive index of 1.33) is infiltrated into a visible PSi resonant waveguide sensor. Water is dropped onto the waveguide top surface and allowed to infiltrate for several minutes. Then the sample is tilted to drain the excess water. Any remaining water droplets that are observed on the surface are removed using optical wipes. Figure 6 shows the experimentally measured waveguide resonance shift of about 2° due to water infiltration. Simulations based on the Bruggeman effective medium approximation [10] indicate that the waveguide resonance shifts approximately 1.5° when water replaces air in the PSi layers. If it is assumed that the air gap above the waveguide is also completely filled with water, the expected resonance shift would be 2.75°. If water does not infiltrate into the pores and only remains on surface, the expected resonance shift is 1.4°. Therefore, it can be concluded that water does penetrate into the pores, although a small volume of water remains on the surface, and is detected using the PSi waveguide sensor. In addition, the quality factor of the resonance decreases because the refractive index in both PSi layers increases due to water infiltration so the thickness of waveguide layer is no longer optimum. The issues of residual liquid on the surface and a decrease in the quality factor of the waveguide resonance will not be significant when using the PSi waveguide sensor to detect small concentrations of biomolecules. Since the biomolecules will be bound inside the pores, vigorous rinsing and drying procedures will be carried out. Moreover, the refractive index change due to the binding of biomolecules will be smaller than the index change due to water so the degradation in the quality of the resonance will be minimal.

![Fig. 6: Waveguide resonance shift due to water infiltration.](image)

![Fig. 7: Waveguide resonance shift due to binding of one monolayer of DNA in the porous silicon waveguide.](image)

**DNA binding**

The waveguide resonance shift due to DNA binding is simulated following [14]. Pores with an average diameter of 20nm and a binding layer of 1nm attached to the pore walls are assumed. The binding layer represents a monolayer of DNA molecules, assumed to have a refractive index of approximately 1.5, which is similar to that of the surface functionalization chemicals aminopropyltriethoxysilane and glutaraldehyde [14]. The effective refractive index of the pore is calculated as a weighted average proportional to the occupied volume of two components: air and the DNA monolayer. The effective refractive index of each PSi layer can then be calculated from the standard Bruggeman approximation [10] and the resonance angle can be determined.
Figure 7 shows the simulated resonance shift as a function of waveguide layer thickness for one monolayer of DNA coverage. Near 300 nm, the waveguide resonance shift is ~0.05°. Since the IR resonant waveguide sensor has a resonance width of 0.1°, the monolayer of DNA can be detected. Preliminary experiments have been performed that demonstrate the ability of PSi waveguide sensors to detect 24-base pair DNA oligos.

CONCLUSION

PSi resonant waveguide sensors have been fabricated and their resonances measured and compared. Best resonance widths have been obtained using both visible and IR light. As the sensor is infiltrated with biomolecules, the resonance angle shifts due to an effective refractive index change in the PSi waveguide. The sensitivity is determined by the resonance width and the magnitude of the resonance shift is affected by the magnitude of the refractive index change due to the presence of the infiltrated target material inside the PSi waveguide. Currently, the resonance width is about 0.1° at 1550nm. A large resonance shift due to the infiltration of water into the waveguide was measured and, from simulation, a monolayer coating of DNA bound to the pore walls can be detected by the resonant waveguide sensor using 1550nm light. Theory suggests that optimized PSi resonant waveguide sensors will be 60 times more sensitive than conventional SPR sensors.

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REFERENCES