Porous silicon waveguide sensor has been demonstrated for high sensitivity detection of biomolecules. Simulations predict a 60-fold enhancement over conventional SPR technology and initial experimental results using DNA oligos have been performed.

Index Terms—Porous silicon, resonant waveguide, biosensor, DNA detection, high sensitivity

I. INTRODUCTION

The study of DNA hybridization and affinity constants is important for gene expression, drug discovery, environmental monitoring and food safety. Label-free optical biosensors are advantageous because they reduce the complexity involved in the sample preparation process. The detection method for label-free sensors is generally based on a refractive index change that results from affinity binding, such as DNA hybridization. Several affinity biosensors, including surface plasmon resonance (SPR) [1], resonant mirrors [2], fiber optics [3], and planar waveguides [4], utilize optical transduction methods to analyze nucleotide interactions. Porous silicon is a good candidate material for this type of biosensing as it has a large surface area to volume ratio (~100 m²/cm³) [5] [6], which facilitates surface-based interactions of probe and target binding.

Optical sensing with porous silicon for DNA detection has been demonstrated using reflectance shifts of Fabry-Perot fringes from single layer porous silicon films [7] and using photoluminescence shifts of multilayer porous silicon microcavities [8]. When the target DNA infiltrates the porous silicon and binds to the probe DNA, the effective refractive index of the porous silicon changes. This effect is due to the small pore size (few-10’s nm), which allows porous silicon to act as an effective medium.

A resonant porous silicon waveguide sensor has recently been proposed to achieve higher sensitivity detection of biomolecules [9]. The porous silicon waveguide sensor has the advantage of being both a thin and resonant structure, which minimizes the infiltration time and requisite volume of the target material as well as provides a sharp resonant feature for which small changes in the optical spectrum can be more easily detected. Furthermore, when compared to other thin resonant biosensors, such as SPR sensors, the porous silicon waveguide biosensor has the advantage of confining the optical energy exactly in the layer where the biomolecules are immobilized and the binding takes place. This leads to a substantial increase in sensitivity of the porous silicon waveguide sensor as compared to SPR sensors and other waveguide sensors for which the optical wave is attenuating as it penetrates the biomolecules [4][10]. Rigorous theoretical calculations indicate that the resonant porous silicon sensor should show a 60-fold improvement in sensitivity over conventional SPR technology [9].

II. SENSOR FABRICATION

The porous silicon waveguide is fabricated by electrochemical etching of p⁺ (0.01Ω·cm) silicon wafers in a 15% ethanoic hydrofluoric acid electrolyte. By applying different current densities during etching, it is possible to control the porosity and thickness of distinct porous silicon layers. Porous silicon waveguides consist of two thin film porous silicon layers, as shown in Figure 1. The first layer (lower porosity) is etched at 5 mA/cm² for 60 seconds and then the applied current is changed to 48 mA/cm² for 53 seconds to form the second layer (higher porosity). The low and high porosity layers have porosities of 52% and 76%, and thicknesses of 300 nm and 1333 nm, respectively. Based on the Maxwell-Garnett effective medium approximation for cylinders, the refractive index of the low porosity layer is 2.167 and the refractive index of the high porosity layer is 1.618 at 1550 nm [11]. Above the porous silicon layers is an air gap. Therefore, as shown in Figure 1, total internal reflection at both interfaces of the top porous silicon layer, or waveguide layer, is possible.

After anodization, the waveguide is oxidized at 900°C for 10 minutes in order to lower the waveguide loss [12] and to prepare the surface for subsequent surface chemistry functionalization procedures. Figure 2 shows the waveguide resonance as measured by a Metricon 2010 prism coupler (prism refractive index of 2.125) after oxidation using 1550 nm light. An evanescent wave with the proper wave vector couples into a waveguide mode when a prism is placed in close proximity to the porous silicon waveguide layer and light is incident at the proper angle. The resonance width is about 0.1°. The substrate mode, which can propagate into the substrate layer, is shown at smaller angle.
III. DNA EXPERIMENTS

From simulation [13], a monolayer coating of DNA bound to the pore walls causes a resonance shift of \( \sim 0.05 \) \( \degree \), which can be detected by the resonant waveguide sensor shown in Figure 2. As a proof of principle experiment, 1000 \( \mu \text{M} \) concentration of 24-base pair DNA oligos is exposed to the porous silicon waveguide sensor. The actual sensitivity of the sensor is believed to be far below this concentration and will be reported in a future communication. The experimental procedure for surface functionalization and DNA binding is as follows. After oxidation, the samples are silanized with 2\% 3-aminopropyltrimethoxysilane in (1:1) DI water and methanol for 20 minutes. The samples are then rinsed with DI water and baked at 100\( \degree \text{C} \) for 10 min. Next, 2.5\% glutaraldehyde in 20 mM HEPES buffer is applied to the sample for 30 min, followed by a 10 min soak in HEPES buffer, rinsing with HEPES buffer, and drying with nitrogen. The sensors are then exposed to probe DNA oligos of concentration 100 \( \mu \text{M} \) (enough to cover all binding sites) in HEPES buffer, rinsing with HEPES buffer, and drying with nitrogen. The sensors are then exposed to probe DNA oligos of concentration 100 \( \mu \text{M} \) (enough to cover all binding sites) in HEPES buffer, incubated for 1 hour, followed by a 20 min soak in buffer, rinsing with buffer, and drying with nitrogen. Finally, target DNA oligos of concentration 1000 \( \mu \text{M} \) are exposed to the waveguide for 1 hour, followed by soaking and rinsing in buffer and drying with nitrogen. Figure 3 shows the waveguide resonance after each preparation step and DNA hybridization. The resonance at the smallest angle is measured after silanization. The adjacent resonances at higher angles are after glutaraldehyde binding, probe DNA, and target DNA. Three measurements are taken after glutaraldehyde binding and five measurements are taken after probe and target DNA binding. Multiple measurements are taken to reduce random errors introduced by the rotary table in the Metricon prism coupler. The maximum error is found to be \( \pm 0.01 \) \( \degree \). TABLE I shows the resonance angles after each functionalization step and after DNA hybridization. Asterisks represent the average value after multiple measurements. The standard deviation for multiple measurements is also shown. For 1000 \( \mu \text{M} \) of target DNA, the resonance shift is 0.18\( \degree \), easily detected by a 0.1\( \degree \) waveguide resonance. A series of control experiments with partial mismatch DNA are currently being carried out along with experiments using lower concentration DNA oligos.

IV. CONCLUSION

Porous silicon resonant waveguides have been fabricated as label-free biosensors. The transduction mechanism is a
resonance angle shift that occurs when infiltrated biomolecules cause an effective refractive index change of the porous silicon waveguide. The detection of 1000 μM DNA is demonstrated as a proof of principle experiment. Further experiments are in progress to determine the actual limits of the porous silicon waveguide sensor sensitivity. The porous silicon waveguide sensor is expected to outperform other affinity-based sensors due to its small size and ability to create a large overlap of the maximum electromagnetic field strength and biomolecule location.

REFERENCES


