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A size selective porous silicon grating-coupled Bloch surface and sub-surface wave biosensor

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ABSTRACT

A porous silicon (PSi) grating-coupled Bloch surface and sub-surface wave (BSW/BSSW) biosensor is demonstrated to size selectively detect the presence of both large and small molecules. The BSW is used to sense large immobilized analytes at the surface of the structure while the BSSW that is confined inside but near the top of the structure is used to sensitively detect small molecules. Functionality of the BSW and BSSW modes is theoretically described by dispersion relations, field confinements, and simulated refractive index shifts within the structure. The theoretical results are experimentally verified by detecting two different small chemical molecules and one large 40 base DNA oligonucleotide. The PSi-BSW/BSSW structure is benchmarked against current porous silicon technology and is shown to have a 6-fold higher sensitivity in detecting large molecules and a 33% improvement in detecting small molecules. This is the first report of a grating-coupled BSW biosensor and the first report of a BSSW propagating mode.

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1. Introduction

Optical biosensors offering label-free detection of relevant chemical species with high sensitivity, rapid read-out, and low-cost are essential for applications spanning medical diagnostics, food safety, and homeland security. For detection of specific label-free bioanalytes and small molecules, plasmonic and photonic sensing platforms have emerged as powerful tools (Adhikari and Majumdar, 2004; Homola et al., 2005; Jane et al., 2009; Kang et al., 2012; Maxwell et al., 2002). In these platforms, the immobilization of an analyte creates a measureable change in the optical properties of the structure, often due to a change in refractive index at the surface of the sensor that is probed by an evanescently decaying field. Because conventional optical platforms utilize planar solids, the total number of available surface binding sites for analytes is limited and there is only minor interaction of the optical field and analyte at the bulk surface, resulting in limited sensitivity. Furthermore, sensors based on planar solid materials are unable to size selectively filter unwanted material or size selectively detect target molecules.

Porous sensing structures address many of the aforementioned challenges and are increasingly being incorporated into optical sensing platforms (Feng et al., 2011; Jane et al., 2009; Jones and Porter, 1988). Porous silicon (PSi) is a particularly attractive label-free biosensing platform due to its highly tunable optical properties, enhanced surface area, and rapid and cost-effective fabrication. The large surface area, arising from the presence of nanoscale pores, allows for improved sensitivity to biomolecule interactions, while the tunable pore dimensions ($\sim$2 nm to $>$100 nm) enable size-selective detection and filtration of larger contaminant species (Lawrie et al., 2010). Label free sensing can be performed through optical transduction schemes and has been demonstrated in a wide variety of PSi optical structures such as single-layer and double-layer interferometers (Dancil et al., 1999; Pacholski et al., 2005), Bragg mirrors (Snow et al., 1999), rugate filters (Li et al., 2012), microcavities (Chan et al., 2000), waveguides (WG) (Rong et al., 2008; Wei et al., 2012; Wei and Weiss, 2011), Bloch surface wave (BSW) structures (Guillermain et al., 2007; Jamois et al., 2010; Michelotti et al., 2010; Qiao et al., 2010), and diffraction gratings (Ryckman et al., 2010). These PSi optical structures have been demonstrated to perform high sensitivity and label-free detection of small proteins, DNA, and other biologically important analytes.

One key limitation facing PSi sensors is the ability to effectively detect both small molecules that can easily infiltrate the porous matrix and large molecules that either slowly diffuse into or are filtered out by the pores. Among the demonstrated porous sensor platforms, the BSW structure holds great promise for sensitively detecting large molecules on the surface of the sensor. The BSW is an optical surface state characterized by distinct field confinement at the interface between a finite 1D photonic crystal, or Bragg mirror, and an external semi-infinite medium (Guillermain et al., 2007;

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Liscidini and Sipe, 2009; Qiao et al., 2010). The BSW is often considered to be the dielectric analog to the surface plasmon polariton (SPP), which provides a strong propagating field at the dielectric-metal interface due to the surface electron oscillation (Descrovi et al., 2010; Liscidini et al., 2009). With the SPP, however, waves propagate along a lossy gold or silver surface and thus have limited propagation lengths and resonant quality factors, considerably lower than what can be achieved with all-dielectric BSW designs (Descrovi et al., 2010; Liscidini et al., 2009; Sinibaldi et al., 2012). Thus, the BSW has emerged as a promising sensing platform for surface-bound molecule detection and has been experimentally demonstrated in various material platforms (Descrovi et al., 2010; Konopsky and Alieva, 2007; Pirotta et al., 2013; Qiao et al., 2010; Toma et al., 2013). Through the use of a porous dielectric, the accessible surface area of a BSW structure can be increased significantly, which promotes improved sensitivity toward small molecules (Descrovi et al., 2010; Guillermain et al., 2007; Jamois et al., 2010; Konopsky and Alieva, 2007; Liscidini et al., 2009; Michelotti et al., 2010; Paeder et al., 2011; Qiao et al., 2010).

In this article, we present a label-free method for size-selective sensing of both small (<2 nm) and large (~ 10 nm) low molecular weight molecules (<15 kDa) using grating-coupled BSW and Bloch sub-surface wave (BSSW) optical modes of a PSi multilayer. The BSW is confined at the multilayer–air interface, which is advantageous for high sensitivity, rapid detection of larger molecules that have difficulty infiltrating the porous matrix. The BSSW, first introduced in this work, is confined just beneath the surface and has a strong sensitivity to small molecules that penetrate the porous matrix. By utilizing a one-dimensional integrated grating at the surface of the structure, coupling can be achieved by far field excitation at a unique resonant angle (Ryckman et al., 2010; Wei and Weiss, 2011) while also eliminating the need for a bulky prism and air gap tuning (Rong et al., 2008). The grating-coupled PSi-BSW/BSSW platform is benchmarked against a grating-coupled PSi-WG (Wei and Weiss, 2011) and demonstrates a larger sensitivity to both small and large molecules.

2. Materials and methods

2.1. Fabrication of BSW/BSSW and WG sensors

The porous silicon structures were fabricated by electrochemical etching of p+ ($\sim 0.01\ \Omega$ cm) Si(1 0 0) wafers. By placing the silicon in a solution containing 15% HF in ethanol and varying the applied current density and duration, the resulting PSi layer thickness and porosity can be tuned. Table 1 shows the current densities and times used to fabricate the structures used in this work. A two second etch break (no current applied) is employed between the formation of each layer. The etching parameters were chosen based on rigorous coupled wave analysis (RCWA) simulations to yield structures with clear optical resonances (Moharam et al., 1995). The etched samples were placed in a 1.5 mM L^{-1} KOH in ethanol solution for 5 min to widen the pores for enhanced molecule infiltration. The samples were then thermally oxidized in air ambient at 500 °C for 5 min. The nominal refractive index of each layer is approximated by fitting the measured reflectivity spectrum of the layer with a thin film simulation that incorporates the scanning electron microscopy (SEM) measured thickness of the layer. For the BSW/BSSW sensor, reflectance spectra of the entire structure were used to fit the multilayer refractive index gradient that gives rise to the BSSW mode. The refractive index gradient was linearly varied from $n_{i1}=1.62$–1.72 and $n_{i2}=1.22$–1.26 for the low current density and high current density layers, respectively. We found that our combination of standard etching, brief KOH treatment, and oxidation produces a natural and reproducible gradient in the optical properties of the multilayer, although the necessary refractive index profile could also be formed by appropriately tuning the PSi etching conditions. We postulate that the dominant contribution to the natural gradient refractive index arises from the diffusion time of the KOH and the duration of KOH exposure to the PSi membrane. This hypothesis is supported by additional tests performed on BSW/BSSW samples with different KOH exposure times for which the BSSW resonance was either not visible or shifted to different angles.

The grating couplers were fabricated by electron beam lithography (EBL). ZEP 520A resist was spun at 6000 rpm to create a ~ 300 nm thick layer and then patterned using a Raith eLine EBL tool. The WG and BSW couplers have grating pitches of $\Lambda_{WG}=1590$ nm and $\Lambda_{BSW/BSSW}=1820$ nm, respectively. After development, the grating duty cycles were measured to be approximately 66% (air: silicon).

Table 1
Electrochemical etching parameters used for device fabrication.

Structure	Layer	Current density (mA/cm^2)	Time (S)	Refractive index	Thickness (nm)
BSW/ BSSW	d_{01}	5	34	Gradient from 1.62 to 1.78	175
	d_{i1} for $i=1$ to 10	5	63		340
	d_{i2} for $i=0$ to 10	48	22	Gradient from 1.22 to 1.26	580
WG	d_{wg}	5	65	1.79	345
	$d_{cladding}$	48	53	1.24	1700

2.2. Chemical preparation

Each sample was soaked in a 4% 3-aminopropyltriethoxysilane (3-APTES) solution in methanol and water for 20 min, followed by a soak in methanol for 10 min to remove the excess silane. Although 3-APTES multilayers are difficult to prevent (Zhu et al., 2012), the 10 min methanol soaking procedure enables a consistent attachment of the molecule. The samples were then thermally annealed at 100 °C for 10 min to promote cross linking between the surface silicon oxide and amino group. A 2.5 $mg \cdot mL^{-1}$ Sulfosuccinimidyl-4-(*N*-maleimidomethyl)cyclohexane-1-carboxylate (Sulfo-SMCC) in 4-(2-hydroxyethyl)-1-piperazineethanesulfonic acid (HEPES) buffer solution was incubated with the samples for 2 h at room temperature, followed by 1 h soak in HEPES solution to remove excess material. Then, 100 μM DNA, (5′-TAG-CTA-TGG-TCC-TCG-TTA-GCT-ATG-GAA-TTC-CTC-GTA-GGC-C 3′), was prepared in HEPES buffer and then mixed with the reducing agent, TCEP (tris(2-carboxyethyl)phosphine) (Pierce) in a 1:1 mixture of methanol and water. The samples were soaked in the 50 μM DNA mixture for 1 h, followed by a 30 min soak in HEPES buffer to remove unattached oligos.

2.3. Measurement technique

The BSW and WG structures were measured between exposure to the solutions discussed in Section 2.2 using a Metricon Model 2010/M Prism Coupler. The prism was removed to allow for far-field coupling. A 1550 nm laser was used as the light source in the variable angle monochromatic fringe observance (VAMFO) mode. A depiction of the setup is illustrated elsewhere (Wei and Weiss 2011).

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