

Investigation of Relationship between Reflection Resonance and Applied Current Density in Bragg Photonic Crystal

Bumseok Kim[†]

Abstract

Relationship between reflection resonance and applied current density in Bragg photonic crystal has been investigated. Multiple bit encodes of distributed Bragg reflector features have been prepared by electrochemical etching of crystalline silicon by using various square wave current densities. Optical characterization of multi-encoding of distributed Bragg reflectors on porous silicon was achieved by Ocean optics 2000 spectrometer for the search of possible applications of multiple bit encoding of distributed Bragg reflectors such as multiplexed assays and chemical sensors. The morphology and cross-sectional structure of multi-encoded distributed Bragg reflectors was investigated by field emission scanning electron micrograph.

Key words : Encoding, Bragg, Photonics, Porous Silicon

1. Introduction

Multistructured porous silicon (PSi) such as distributed Bragg reflectors (DBR) PSi and Rugate PSi have been a topic of interest, because of their use in chemical and biological sensors, biomaterials, and optical devices^[1-4]. They exhibit unique optical properties providing the reflection of a specific wavelength in the optical reflectivity spectrum and are an attractive candidate for building nanostructured composite materials^[5]. DBR PSi is typically prepared by an applying a computer-generated pseudo-square current waveform to the etch cell which results two distinct indices and exhibits photonic structure of Bragg filters^[6-10].

Rugate-structured porous silicon having photonic structure of rugate filter in which refractive index varies sinusoidally has been recently developed by applying a computer-generated pseudo-sinusoidal current waveform^[11]. Chemical modification of PSi multilayer exhibits the modification of its physical, chemical, and electronic properties. Chemical or bio molecule can be detected based on changes in the spectral interference

pattern^[12]. Biosensor based on porous silicon interferometer has a great advantage due to a large surface area matrix for immobilization of a variety of biomolecules such as enzymes^[13], protein^[14], and DNA fragments^[15]. PSi is biocompatible and bioresorbable; it has tunable pore sizes and volumes, a high surface area, and unique optical properties that allow in-vivo monitoring. A unique attribute of PSi is its ability to optically report on the loading of a molecule within the porous nanostructure.

Sailor et al. recently reported that the electronic or optical properties of mono-layered PSi can also be used as the transducer of biomolecular interaction in biosensor application^[16,17]. The prerequisite for using porous silicon as an optical interferometric biosensor is to adjust the size as well as the geometrical shape of the pores by choosing the appropriate etching parameters. The pore size has to be large enough to allow biomolecules to enter the pores freely but small enough to retain optical reflectivity of the porous silicon surface. Moreover, it is necessary that the material be mechanically and chemically stable in aqueous solutions to provide reproducible and predictable binding signals.

Here we report that the investigation of relationship between reflection resonance and applied current density in Bragg photonic crystal and a method of preparing one-dimensional photonic crystals showing multiple

Department of Chemistry, Chosun University, 375 Seosuk-dong, Gwangju 501-759, Korea

[†]Corresponding author : afewkim@gmail.com
(Received : February 7, 2012, Revised : March 25, 2012,
Accepted : March 27, 2012)

Bragg features in their optical reflectivity spectrum.

2. Experimental Section

2.1. Sample Preparation

Porous silicon samples were prepared by electrochemical etching of heavily doped p⁺-type silicon wafers (boron doped, polished on the (100) face, resistivity of 0.8~1.2 mΩ-cm, Siltronix, Inc.). The etching solution consisted of a 3 : 1 volume mixture of aqueous 48% hydrofluoric acid (ACS reagent, Aldrich Chemicals) and absolute ethanol (ACS reagent, Aldrich Chemicals). The galvanostatic etch was carried out in a Teflon cell by using a two-electrode configuration with a Pt mesh counter electrode. Five alternating etch currents between two different values used: (a) a high current of 50 mA/cm² for 3 sec and a low current of 5 mA/cm² for 100 sec with 20 repeats, (b) a high current of 100 mA/cm² for 3 sec and a low current of 10 mA/cm² for 100 sec with 20 repeats, (c) a high current of 150 mA/cm² for 3 sec and a low current of 15 mA/cm² for 100 sec with 20 repeats, (d) a high current of 200 mA/cm² for 3 sec and a low current of 20 mA/cm² for 100 sec with 20 repeats, (e) a high current of 250 mA/cm² for 3 sec and a low current of 25 mA/cm² for 100 sec with 20 repeats. The anodization current was supplied by a Keithley 2420 high-precision constant current source controlled by a computer to allow the formation of multi-Bragg-featured photonic crystals. To prevent the photogeneration of carriers, the anodization was performed in the dark. After formation, the samples are rinsed with pure ethanol and dried with nitrogen gas. The entire electrochemical process is carried out under constant current supplied by a computer controlled Keithley 2420 power source meter.

2.1. Instrumentation and Data Acquisition

Optical reflectivity spectra are measured using a tung-

sten halogen lamp and an Ocean Optics S2000 CCD spectrometer fitted with a fiber optic input. The reflected light collection end of the fiber optic is positioned at the focal plane of the optical microscope. The SEM images for the morphology of porous silicon were obtained by using a cold-field emission scanning electron microscope (FE-SEM, S-4700, Hitachi).

3. Result and Discussion

A DBR PSi exhibits a high reflectivity band with a Bragg wavelength λ_{Bragg} , depending on the thickness of the layers (d_1 , d_2) and the corresponding refractive indices (n_1 , n_2). The m th order of the Bragg peak is given by:

$$m\lambda_{\text{Bragg}} = 2(d_1 \cdot n_1 + d_2 \cdot n_2)$$

Typical etch parameters for DBR PSi structure involves using a periodic square wave current between low and high current densities.

Schematic diagram for a method of preparing one-dimensional photonic crystals showing multiple Bragg features in their optical reflectivity spectrum was shown in Fig. 1. To obtain the multiple Bragg reflection, the relationship between reflection resonance and an applied current density during an etching process was investigated. An amplitude of applied current density from the Bragg condition increased by 2-fold.

Sample prepared by using an etching condition of a high current of 50 mA/cm² for 3 sec and a low current of 5 mA/cm² for 100 sec with 20 repeats and by applying a computer-generated square current waveform were displayed single high reflection resonance having many sidelobes at the wavelength of 580 nm and showed in Fig. 2. Photonic crystals displaying Bragg structure result in a mirror with high reflectivity in a specific narrow spectral region.

Sample prepared by using an etching condition of a

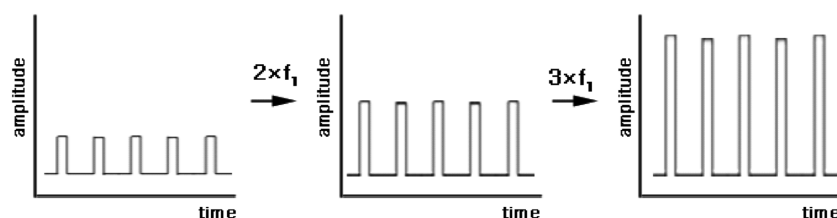


Fig. 1. Schematic diagram for the generation of multiple DBR peaks.

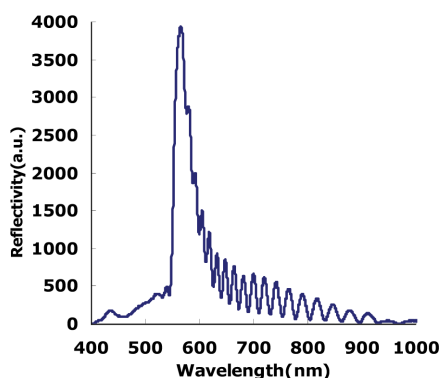


Fig. 2. Reflection spectrum of porous silicon showing single DBR peak.

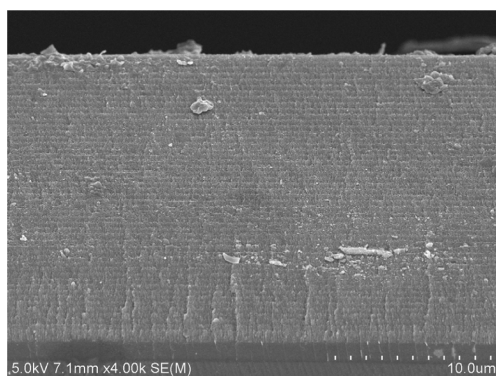


Fig. 3. FE-SEM images of PSi samples prepared with an etching condition of a high current of 50 mA/cm^2 for 3 sec and a low current of 5 mA/cm^2 for 100 sec with 20 repeats.

high current of 50 mA/cm^2 for 3 sec and a low current of 5 mA/cm^2 for 100 sec with 20 repeats and by applying a computer-generated square current waveform were displayed single high reflection resonance having many sidelobes at the wavelength of 580 nm and showed in Fig. 2. Photonic crystals displaying Bragg structure result in a mirror with high reflectivity in a specific narrow spectral region.

Cross-sectional SEM images of PSi prepared by using a periodic square wave current between low and high current densities shown in Fig. 3 was obtained using a cold field emission scanning electron microscope (FE-SEM, S-4800, Hitachi). The cross-sectional image of DBR PSi shown illustrates that the multilayer DBR PSi has a depth of a few microns. A repeating etch process results in two distinct refractive indices. The resulting porous silicon film exhibited a porosity depth

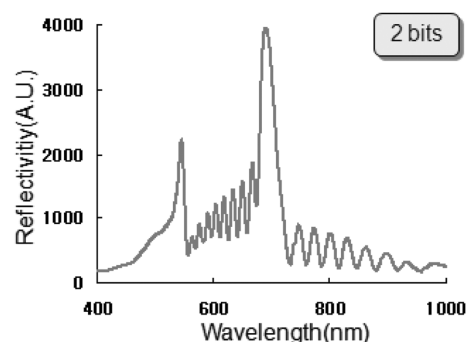


Fig. 4. Reflection spectrum of porous silicon sample etched with a high current of 100 mA/cm^2 for 3 sec and a low current of 10 mA/cm^2 for 100 sec with 20 repeats.

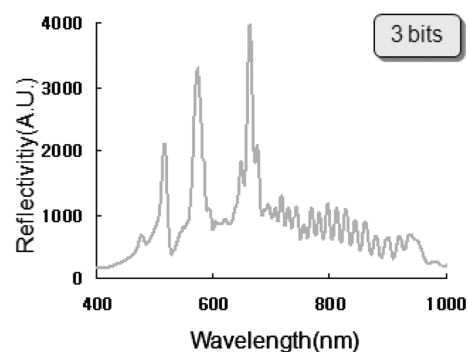


Fig. 5. Reflection spectrum of porous silicon sample etched with a high current of 150 mA/cm^2 for 3 sec and a low current of 15 mA/cm^2 for 100 sec with 20 repeats.

profile that related directly to the current-time profile used in the etch.

An amplitude of applied current density used for sample 1 increased by 2 times. A high current of 100 mA/cm^2 for 3 sec and a low current of 10 mA/cm^2 for 100 sec with 20 repeats were used. Resulting porous silicon displayed double reflection peaks at the wavelength of 550 and 680 nm in the reflection spectra as shown in Fig. 4. An amplitude of applied current density used for sample 1 increased by 3 times which was a high current of 150 mA/cm^2 for 3 sec and a low current of 15 mA/cm^2 for 100 sec with 20 repeats. Porous silicon sample 3 showed three reflection bands at the wavelength of 550, 580, and 680 nm in the reflection spectra as shown in Fig. 5. The reflection peaks were not placed in the same physical locations obtained from sample 2.

An amplitude of applied current density used for sample 1 increased by 4 times which was a high current of

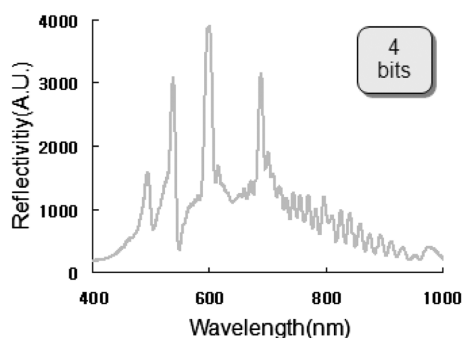


Fig. 6. Reflection spectrum of porous silicon sample etched with a high current of 200 mA/cm² for 3 sec and a low current of 20 mA/cm² for 100 sec with 20 repeats.

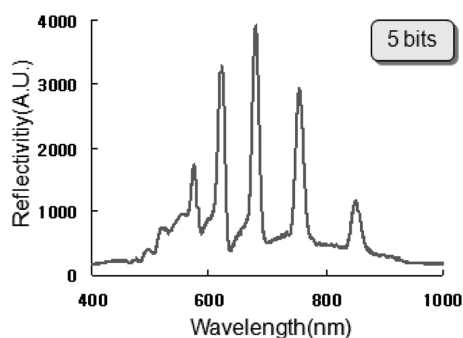


Fig. 7. Reflection spectrum of porous silicon sample etched with a high current of 250 mA/cm² for 3 sec and a low current of 25 mA/cm² for 100 sec with 20 repeats.

200 mA/cm² for 3 sec and a low current of 20 mA/cm² for 100 sec with 20 repeats. Porous silicon sample 4 showed four reflection bands at the wavelength of 480, 520, 600, and 680 nm in the reflection spectra as shown in Fig. 6.

The reflectance spectra of five encoded porous silicon samples were obtained and showed five-bit encoding. An amplitude of applied current density used for sample 1 increased by 5 times which was a high current of 250 mA/cm² for 3 sec and a low current of 25 mA/cm² for 100 sec with 20 repeats. Porous silicon sample 5 showed five reflection bands in the reflection spectra as shown in Fig. 7. The wavelengths of the spectral peaks were not controllable by changing the etch parameters, but this could be useful for encoding information.

4. Conclusion

Photonic crystals displaying multi Bragg structure

were prepared by applying a computer-generated square current waveform. Five individual etched samples were prepared by using different etch parameters. As an amplitude of applied current density from the Bragg condition increased by 2-fold, the number of reflection bands increased. The reflection peaks were not placed in the same physical locations for each samples, but this encoded porous silicon could be useful for encoding information.

References

- [1] H. Sohn, S. Letant, M. J. Sailor and W. C. Trogler, "Detection of Fluorophosphonate Chemical Warfare Agents by Catalytic Hydrolysis with a Porous Silicon Interferometer", *J. Am. Chem. Soc.*, Vol. 122, p. 5399, 2000.
- [2] C. Pacholski, M. Sartor, M. J. Sailor, F. Cunnin and G. M. Miskelly, "Biosensing Using Porous Silicon Double-Layer Interferometers: Reflective Interferometric Fourier Transform Spectroscopy", *J. Am. Chem. Soc.*, Vol. 127, p. 11636, 2005.
- [3] L. T. Canham, M. P. Stewart, J. M. Buriak, C. L. Reeves, M. Anderson, E. K. Squire, P. Allcock and P. A. Snow, "Derivatized Porous Silicon Mirrors: Implantable Optical Components with Slow Resorbability", *Phys. Status Solidi.*, Vol. 182, p. 521, 2000.
- [4] V. Lehmann, R. Stengl, H. Reisinger, R. Detemple and W. Theiss, "Optical shortpass filters based on macroporous silicon", *Appl. Phys. Lett.*, Vol. 78, p. 589, 2001.
- [5] Y. Y. Li, F. Cunin, J. R. Link, T. Gao, R. E. Betts, S. H. Reiver, V. Chin, S. N. Bhatia, and M. J. Sailor, "Polymer Replicas of Photonic Porous Silicon for Sensing and Drug Delivery Applications", *Science*, Vol. 299, p. 2045, 2003.
- [6] A. Janshoff, K. P. S. Dancil, C. Steinem, D. P. Greiner, V. S. -Y. Lin, C. Gurtner, K. Motesharei, M. J. Sailor, and M. R. Ghadiri, "Macroporous p-Type Silicon Fabry-Perot Layers. Fabrication, Characterization, and Applications in Biosensing", *J. Am. Chem. Soc.*, Vol. 120, p. 12108, 1998.
- [7] B. J. Lee, S. G. Kim and H. Sohn, "Optically encoded smart dust from DBR porous silicon", *Key Eng. Mater.*, Vol. 321, p. 53, 2006.
- [8] M. Cazzanelli, C. Vinegoni, and L. Pavesi, "Temperature dependence of the photoluminescence of all-porous-silicon optical microcavities", *J. Appl. Phys.*, Vol. 85, p. 1760, 1999.
- [9] V. Lehmann, R. Stengl, H. Reisinger, R. Detemple,

- and W. Theiss, "Optical shortpass filters based on macroporous silicon", *Appl. Phys. Lett.*, Vol. 78, p. 589, 2001.
- [10] C. Mazzoleni and L. Paves, "Application to optical components of dielectric porous silicon multilayers", *Appl. Phys. Lett.*, Vol. 67, p. 2983, 1995.
- [11] J. Park, S. Cho, Y. C. Ko, H. J. Sohn (2007) *Korean Phys. Soc.*, 50, 695.
- [12] A. Janshoff, K.-P. S. Dancil, C. Steinem, D. P. Greiner, V. S.-Y. Lin, C. Gurtner, K. Motesharei, M. J. Sailor, M. R. Ghadiri, *J. Am. Chem. Soc.* 1998, 120, 12108.
- [13] J. Drott, K. Lindstrom, L. Rosengren and T. Laurell, "Porous silicon as the carrier matrix in microstructured enzyme reactors yielding high enzyme activities", *J. Micromech. Microeng.*, Vol. 7, p. 14, 1997.
- [14] T. Laurell, J. Drott, L. Rosengren and K. Lindstrom, "Enhanced enzyme activity in silicon integrated enzyme reactors utilizing porous silicon as the coupling matrix", *Sensor. Actuat. B. Chem.*, Vol. 31, p. 161, 1996.
- [15] K. L. Beattie, W. G. Beattie, L. Mengm, S. L. Turner, R. Coral- Vazquez, D. D. Sith, P. M. McIntyre, D. D. Dao, "Advances in genosensor research", *Clin. Chem.*, Vol. 41, p. 700, 1995.
- [16] V. S. Y. Lin, K. Motesharei, K. P. S. Dancil, M. J. Sailor and M. R. Ghadiri, "A Porous Silicon-Based Optical Interferometric Biosensor", *Science*, Vol. 278, p. 840, 1997.
- [17] M. J. Sailor, "Properties of Porous Silicon", *Datareview Ser. NO. 18*, Canham: London, p. 364, 1997.