

## Multiple-Bit Encodings of Bragg Photonic-structures by Using Consecutive Etch with Various Square Wave Currents

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### Abstract

New method to encode multiple photonic features of Bragg type reflector on silicon wafer 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

The development of a new technology to build a photonic structure has been of great interest since it is too complicated to fabricate when conventional lithographic methods are used. The materials having photonic features were useful for a variety of applications, such as chemical and biological sensors or medical diagnostics. Since the porous silicon is one of candidates to build a photonic feature, the research on porous silicon has been investigated for possible applications of porous silicon photonic crystals such as a micro chemical reactors, micro fuel cells, and optical band pass filters<sup>[1,2]</sup>.

The advantage of porous silicon is its very high surface area for reactions, its unique photonic properties for optoelectronic devices, and process compatibility with conventional semiconductor technology<sup>[3,4]</sup>. High surface area of porous silicon has been shown to be useful in a variety of analytical applications, in micro electro mechanical systems as a matrix for matrix-assisted laser desorption ionization mass spectroscopy and especially in chemical and biological sensors<sup>[5,6]</sup>.

For studying genomes and identifying candidate drugs, a large number of parallel experiments should be performed in a short period of time. To achieve this purpose, multiplexed assays<sup>[7,8]</sup> are powerful tools<sup>[3]</sup>. Beads or particles containing many independent codes made by using quantum dots, fluorescent molecules, and metal oxide have been recently used for these applications<sup>[9-14]</sup>.

If one can build a nanostructure with complex photonic features, one of the simplest and cheapest methods could be an electrochemical etching of silicon wafers. Porous silicon, one of dielectric materials, shows the unique optical properties as a result of an effective refractive index depending directly on the porosity. Electrochemical etching offers both the opportunity to modulate the porosity in depth and the fabrication of structures with any refractive index profile. The thicknesses, porosities and average pore diameters of porous layers can be controlled by using parameters such as the current density, the duration of etch, cycle, and the composition of the etchant solution<sup>[15]</sup>. The Bragg photonic features can be fabricated by alternating the etch current between two different values, a high and a low current. This method leads to low and high refractive indices, respectively.

Here we report a method of preparing one-dimensional photonic crystals showing multiple Bragg features in their optical reflectivity spectrum.

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## 2. Experimental Section

### 2.1. Sample Preparation

Porous silicon samples were prepared by electrochemical etching of heavily doped p<sup>++</sup>-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. Three alternating etch currents between two different values : (a) a high current of 5 mA/cm<sup>2</sup> for 90 sec and a low current of 50 mA/cm<sup>2</sup> for 3 sec with 20 repeats, (b) a high current of 5 mA/cm<sup>2</sup> for 180 sec and a low current of 50 mA/cm<sup>2</sup> for 6 sec with 20 repeats, (c) a high current of 5 mA/cm<sup>2</sup> for 270 sec and a low current of 50 mA/cm<sup>2</sup> for 12 sec with 20 repeats, were used. 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, was performed the anodization in the dark. After formation, the samples are rinsed with pure ethanol and dried with nitrogen gas.

### 2.1. Instrumentation and Data Acquisition

Optical reflectivity spectra are measured using a tungsten 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

Photonic crystals displaying Bragg structure result in a mirror with high reflectivity in a specific narrow spectral region. These photonic crystals are prepared by applying a computer-generated square current waveform. The Bragg feature exhibits a very sharp line in the optical reflectivity spectrum, which can be tuned to appear anywhere in the visible to near-infrared spectral

range, depending on the programmed etch waveform.

For the preparation of Bragg feature, an alternating etch current, a high current of 5 mA/cm<sup>2</sup> for 90 sec and a low current of 50 mA/cm<sup>2</sup> for 3 sec, with 20 repeats was used. Figure 1 showed the reflection spectrum of Bragg-featured photonic crystal. 560 nm of reflection maximum wavelength was observed.

The SEM images shown in Figure 2 were obtained by using a cold-field emission scanning electron microscope (FE-SEM, S-4700, Hitachi). The cross-sectional

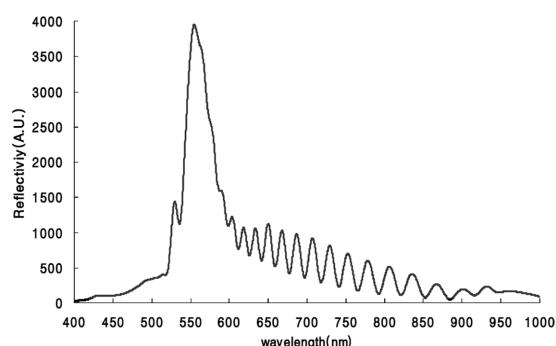


Fig. 1. Reflection spectrum of Bragg-type photonic crystals.

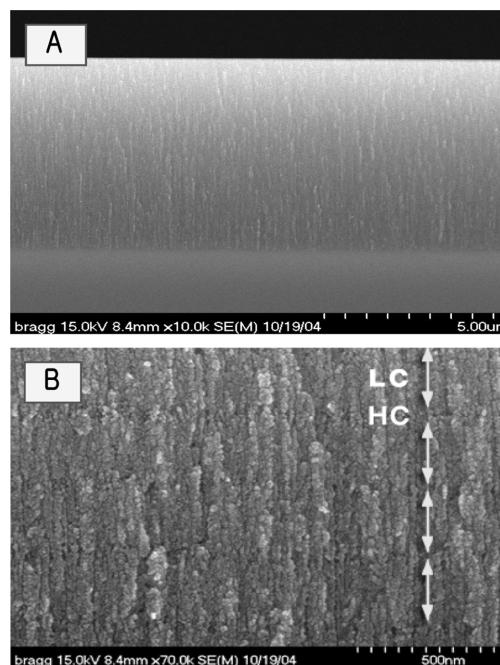


Fig. 2. Cross-sectional SEM images of as-prepared Bragg-type photonic crystals, (a) low magnification, (b) high magnification.

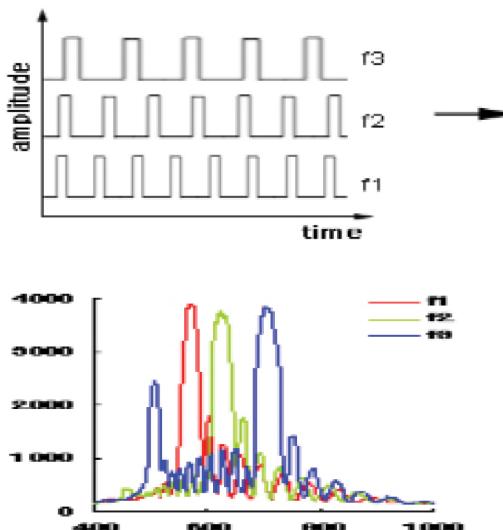


Fig. 3. Reflection spectra of samples prepared by using (f1) a high current of  $5 \text{ mA/cm}^2$  for 90 sec and a low current of  $50 \text{ mA/cm}^2$  for 3 sec with 20 repeats (red), (f2) a high current of  $5 \text{ mA/cm}^2$  for 180 sec and a low current of  $50 \text{ mA/cm}^2$  for 6 sec with 20 repeats (green), and (f3) a high current of  $5 \text{ mA/cm}^2$  for 270 sec and a low current of  $50 \text{ mA/cm}^2$  for 12 sec with 20 repeats (blue).

image illustrated that the sample had profile of two refractive indices with a depth of few microns. Bragg feature possess a varying porosity in the direction perpendicular to the plane of the filter.

Three individual etched samples were prepared by using different etch parameters and shown in Figure 3. Reflection resonances obtained by using etching parameters of f1, f2, and f3 took place at 580, 610, and 710 nm, respectively. Values for a high current and low current were fixed. Etching times and interval times between each etching procedure were increased. As etching times and interval times increased, reflection resonances shifted to the longer wavelength.

Consecutive etching was performed by using two alternating etch currents. Silicon wafer was etched with a high current of  $5 \text{ mA/cm}^2$  for 90 sec and a low current of  $50 \text{ mA/cm}^2$  for 3 sec with 20 repeats. And then consecutive etching was performed with a high current of  $5 \text{ mA/cm}^2$  for 180 sec and a low current of  $50 \text{ mA/cm}^2$  for 6 sec with 20 repeats. Figure 4 showed the reflection spectrum of sample prepared from the consecutive etching with two square current densities. The reflection peaks were not placed in the same physical locations.

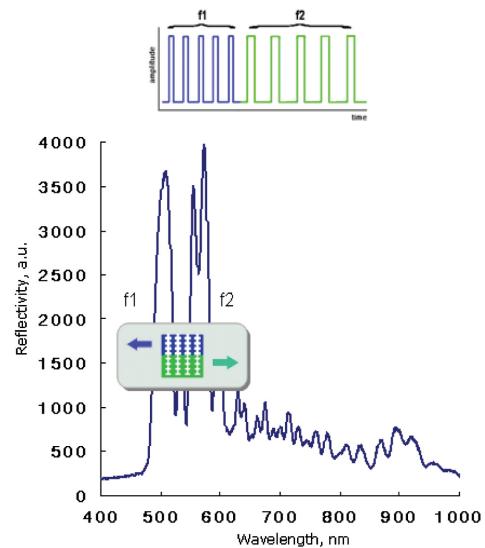


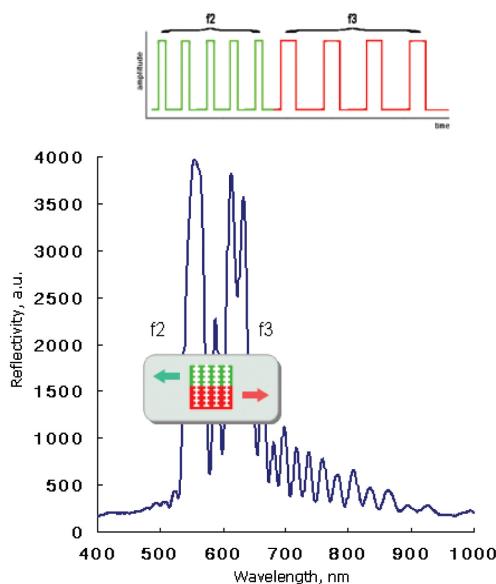
Fig. 4. Reflection spectrum of sample prepared from the consecutive etching with two square current densities. f1 and f2 refer to the etching condition of (a) a high current of  $5 \text{ mA/cm}^2$  for 90 sec and a low current of  $50 \text{ mA/cm}^2$  for 3 sec with 20 repeats and (b) a high current of  $5 \text{ mA/cm}^2$  for 180 sec and a low current of  $50 \text{ mA/cm}^2$  for 6 sec with 20 repeats, respectively.

Photonic feature for the first etching appeared at about 500 nm in the optical reflection spectrum. However, the reflection peaks resulted from second etching appeared at about 560 nm as doublet.

Another consecutive etching was performed. Silicon wafer was etched with a high current of  $5 \text{ mA/cm}^2$  for 180 sec and a low current of  $50 \text{ mA/cm}^2$  for 6 sec with 20 repeats. And then consecutive etching was performed with a high current of  $5 \text{ mA/cm}^2$  for 270 sec and a low current of  $50 \text{ mA/cm}^2$  for 9 sec with 20 repeats.

Figure 5 showed the reflection spectrum of sample prepared and a similar reflection pattern was observed. Photonic feature for the first etching appeared at about 560 nm in the optical reflection spectrum. The reflection peaks resulted from second etching also appeared as doublet at about 530 nm.

According to above results, the reflection peak at shorter wavelength is responsible for first etching procedure and displays single reflection resonance, while the reflection peak at longer wavelength is responsible for second etching procedure and displays double reflection resonances. This doublet may result in the



**Fig. 5.** Reflection spectrum of sample prepared from the consecutive etching with two square current densities. f<sub>2</sub> and f<sub>3</sub> refer to the etching condition of (a) a high current of 5 mA/cm<sup>2</sup> for 180 sec and a low current of 50 mA/cm<sup>2</sup> for 6 sec with 20 repeats and (b) a high current of 5 mA/cm<sup>2</sup> for 270 sec and a low current of 50 mA/cm<sup>2</sup> for 9 sec with 20 repeats, respectively.

interference of former square current during the second etching process.

#### 4. Conclusion

Photonic crystals displaying Bragg structure were prepared by applying a computer-generated square current waveform. The SEM images obtained by using a cold-field emission scanning electron microscope displayed that the cross-sectional image of sample had profile of two refractive indices with a depth of few microns. Bragg feature possess a varying porosity in the direction perpendicular to the plane of the filter.

Three individual etched samples were prepared by using different etch parameters. Etching times and interval times were increased with the fixed high and low currents. As etching times and interval times increased, reflection resonances shifted to the longer wavelength.

Consecutive etching was performed by using two alternating etch currents. The reflection spectrum of sample prepared from the consecutive etching with two square current densities displayed that the reflection

peaks for the first etching appeared singlet but the reflection peaks for the second etching appeared as doublet. This doublet may result in the interference of former square current during the second etching process.

This multiple photonic structure might be useful for multiplexed assays to study genomes and to identify candidate drugs, in which a large number of parallel experiments.

#### References

- [1] S. H. Jang, "Chemical and Physical Properties of Porous Silicon", Journal of the Chosun Natural Science, Vol. 4, No. 1, p. 1, 2011.
- [2] S. G. Kim, "Optical Characterization of Smart Dust Based on Photonic Crystals and Its Sensing Applications", Journal of the Chosun Natural Science, Vol. 4, No. 1, p. 7, 2011.
- [3] Y. D. Koh, "1-D Photonic Crystals Based on Bragg Structure for Sensing and Drug Delivery Applications", Journal of the Chosun Natural Science, Vol. 4, No. 1, p. 11, 2011.
- [4] K. S. Jung, "Fabrication and Characterization of DBR Porous Silicon Chip for the Detection of Chemical Nerve Agents", Journal of the Chosun Natural Science, Vol. 3, No. 4, p. 237, 2010.
- [5] S. D. Cho, "Detection of Nitroaromatic Compounds with Functionalized Porous Silicon Using Quenching Photoluminescence", Journal of the Chosun Natural Science, Vol. 3, No. 4, p. 202, 2010.
- [6] S. H. Jang, "Study on Thickness of Porous Silicon Layer According to the Various Anodization Times", Journal of the Chosun Natural Science, Vol. 3, No. 4, p. 206, 2010.
- [7] S. H. Jang, "Investigation of Relationship between Etch Current and Morphology and Porosity of Porous Silicon", Journal of the Chosun Natural Science, Vol. 3, No. 4, p. 120, 2010.
- [8] D. H. Jung, "Biosensor Based on Distributed Bragg Reflector Photonic Crystals for the Detection of Protein A", Journal of the Chosun Natural Science, Vol. 3, No. 1, p. 33, 2010.
- [9] M. Bruchez, M. Moronne, P. Gin, S. Weiss and, A. P. Alivisatos, "Direct Physical Measure of Conformational Rearrangement Underlying Potassium Channel Gating", Science, Vol. 281, p. 213, 1996.
- [10] J. A. Ferguson, T. C. Boles, C. P. Adams, and D. R. Walt, "A fiber-optic DNA biosensor microarray for the analysis of gene expression", Nat. Biotechnol., Vol. 14, p. 1681, 1996.

- [11] H. Fenniri, L. Ding, A. E. Ribbe, and Y. Zyrianov, "Barcoded Resins: A New Concept for Polymer-Supported Combinatorial Library Self-Deconvolution", *J. Am. Chem. Soc.*, Vol. 123, p. 8151, 2001.
- [12] W. C. W. Chan and S. Nie, "Quantum Dot Bioconjugates for Ultrasensitive Nonisotopic Detection", *Science*, Vol. 281, p. 2016, 1998.
- [13] S. R. Nicewarner-Pena, R. G. Freeman, B. D. Reiss, L. He, D. J. Peña, I. D. Walton, R. Cromer, C. D. Keating, and M. J. Natan, "Submicrometer Metallic Barcodes", *Science*, Vol. 294, p. 137, 2001.
- [14] J. M. Han, "Photoluminescence of Porous Silicon According to Various Etching Times and Various Applied Current Densities", *Journal of the Chosun Natural Science*, Vol. 3, No. 3, p. 148, 2010.
- [15] Y. D. Koh, "Analysis on Oxidation of Porous Silica Obtained from Thermal Oxidation of Porous Silicon", *Journal of the Chosun Natural Science*, Vol. 3, No. 3, p. 153, 2010.