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Fabrication of optically smooth surface on the cleavage of porous silicon by gas cluster ion irradiation

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ABSTRACT

Electrochemical etching of Si is a promising method of fabricating multilayer photonic structures with hundreds of layers. Nevertheless, the natural cleavage of the porous silicon structure has roughness and then high optical scattering that embarrasses the usage of porous silicon in optical devices. In this study, gas cluster ion beam irradiation was suggested as a polishing technique. The bombardment results in surface smoothing and consequent enhancement of light reflection without significant difference in sputtering rate of layers with different porosity. Increasing irradiation dose results in different behavior of porous layers: densification of low-porosity structures and nano-rods formation in high-porosity layers.

1. Introduction

In recent decades, light propagation in optical periodic structures, photonic crystals (PhC), attracts interest of researchers due to unique possibilities to control light. The enhancement of known optical effects like Raman scattering [1], Faraday [2] and Kerr [3] effects and second harmonic generation [4] has been observed as well as existence of new effects like photonic band gap [5] and slow light [6] was discovered in photonic crystals. Some new optical effects require PhCs with high number of periods [7,8] and good surface quality to avoid light scattering. A promising way of producing such structures is electrochemical etching of porous silicon. Periodic modulation of electric current creates porous layers with alternating porosity which opens the possibility to produce structures with thousands of periods [9]. These structures have front surfaces as smooth as original silicon wafer. However, there are a lot of optical effects theoretically predicted since thirty years ago [10] up to now [11] that require high quality of the side facet of one-dimensional photonic crystal. Typically, the side facets of porous silicon PhC are produced by mechanical cleavage of the sample. Unfortunately, the mechanically cleft samples have a rough side surface only because of the porous structure of the samples [12]. The cleavage of a porous structure has defects with size not less than one pore diameter and defects can likely be larger, exceeding 100–500 nm, which is unacceptable for optical applications due to extremely high light scattering. Methods of surface polishing are therefore needed.

Gas cluster ion beams are widely known as an instrument for surface relief smoothing. A vast number or works are dedicated to investigation of cluster ion irradiation effect on the relief of simple and composite materials [13–16]. It is shown that surface roughness can be reduced down to a few angstroms. At the same time, since clusters interact only with the uppermost atomic layers of the target, defects are not generated in the volume of the processed media [17]. Relief evolution of surfaces with initially ordered pattern deserves special attention and is described in Ref. [18]. However, the influence of cluster ion irradiation on nanostructures, such as nano-rods, nano-spheres, etc. was not observed experimentally. This is the first attempt to use gas clusters to obtain smooth surface on porous silicon. Moreover, since sputtering effects on nanostructures evolution are poorly studied theoretically [19,20] and even less studied experimentally [21]. From this point of view we suggest porous silicon, which is consisted of nano-rods separated by a free space, as a good object for such an investigation.

In this work, we have performed gas cluster irradiation of porous silicon samples, and analyzed resulted surface changes by scanning electron microscopy, atomic force microscopy and optical reflectivity measurements. In total, two sets of samples were

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prepared, irradiated and investigated: samples having with 200 nm thick layers, subjected to low fluence irradiation ($5\cdot10^{15}$ ions/cm^2) and other samples with 1000 nm thick layers, subjected to high fluence irradiation ($1.5\cdot10^{16}$ ions/cm^2).

2. Experimental methods

Porous silicon photonic crystal samples were manufactured by electrochemical etching of bulk silicon [9]. Single-crystal p-type Si (001) wafers doped with B with resistivity 0.005 Ohm·cm were processed in an electrolyte containing 21% hydrofluoric acid. The layers with low (65% total pore volume) and high (73% total pore volume) porosity were formed under etching current of 40 and 200 mA/cm^2, respectively. Average pore diameter was estimated by BET (Brunauer–Emmett–Teller [22]) thermo-desorption and equaled 20 nm and 65 nm for the two layers, which is in good agreement with SEM images. Two sets of samples were prepared: with thickness of the layers 200 nm and 250 nm for optical experiments and 800 nm and 1200 nm for AFM experiments. Overall thicknesses of the multilayered structures were more than 100 μm.

Samples irradiation was performed using MSU (Moscow State University) gas cluster ion beam accelerator [23]. Argon clusters were formed in a jet during adiabatic expansion of the gas through a supersonic nozzle. The clusters were extracted with a skimmer, ionized by electron impact and accelerated by an electrostatic potential of 10 keV. Cluster ion size was estimated to be 800 atoms per charge unit at the maximum of the size distribution. Mono-atomic and light cluster ions were deflected by a permanent magnet since the effects of their interaction with targets differs from the massive cluster ones. The irradiation doses were $0.5\cdot10^{16}$ cm^{-2} and$1.5\cdot10^{16}$ cm^{-2}. During the irradiation, a half of each sample was covered with a mask. This allowed comparison of parameters of processed and unprocessed areas within the same sample.

Structure and topography of both areas of each sample were observed by scanning electron microscopy (SEM) and by tapping mode atomic force microscopy (AFM). Measurement of reflectance was performed using an Xe high-pressure light source. The light was focused under 45° incidence angle onto the 200 μm area on the sample cleavage. The reflected light was collected under the mirror angle by a micro-lens into a multimode optical fiber and guided to diffraction spectrometer Avesta ASP100MF. Using an automated actuator, the sample was moved along the cleavage parallel to its plane. The point of measurement thus moved along the lateral sample surface.

3. Results and discussion

Typical SEM images of the pristine and irradiated areas of porous silicon sample cleavage are shown in Fig. 1. Irradiation dose equaled $0.5\cdot10^{16}$cm^{-2}, layer thicknesses 200/250 nm. Such structures are used in photonic applications. From the central part of the figure depicting the border between these areas it is seen that the irradiated surface became more uniform. The number of elements with sharp edges decreased, surface relief became more smooth. Left and right parts of the figure contain larger scale images of irradiated and pristine surfaces, respectively. Dark stripes are the layers with low density, and light stripes are the layers with high density. High density layers were smoothed without significant changes in structure. Low density layers changed their structure: pore walls are distorted; nano-rods with smooth walls are formed. Investigation of a sample face by AFM showed that there are no significant differences in etching rate of the layers under this dose, but rod-like figures of the low-density layer protrude from the surface for a few tens of nanometers.

Spectral dependence of optical reflectance was measured at different spots along the cleavage surface (Fig. 2). Measured reflectance value was normalized to the reflection of an ideal surface calculated by Fresnel equations. Its value depends on the wavelength of the incident light. For shorter wavelengths, reflectance is lower due to strong light scattering on the sample surface. Position 2 mm corresponds to the border between the pristine and irradiated regions. Passing through the border to the irradiated area, reflectivity increases by a factor of two. It confirms that the surface becomes smoother and light dissipation on sharp edges is suppressed. Thus, cluster ion polishing has a positive effect on the optical characteristics of multilayered porous silicon surface.

Fig. 3 shows SEM images of pristine and irradiated regions of porous silicon with the same porosity, but wider layers (800/1200 nm). Such a structure was investigated to decrease the influence of the adjacent layers during sputtering, such as redeposition, and to provide more accurate AFM measurements of

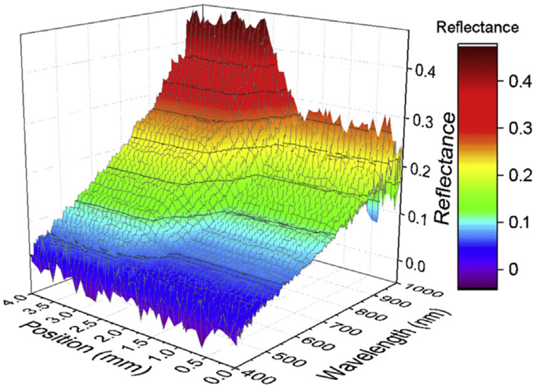

Fig. 2. Spectral dependence of normalized optical reflectivity at different spots along the sample cleavage. Positions 0–2 mm correspond to pristine surface, 2–4 mm correspond to irradiated surface.

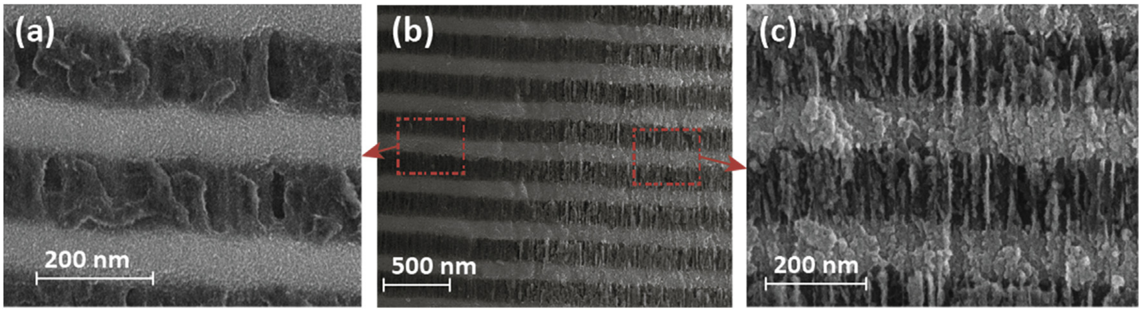

Fig. 1. SEM image of multilayered porous silicon cleavage. (a) – irradiated area, (b) – border between irradiated and pristine areas, (c) – pristine area. Irradiation dose $0.5\cdot10^{16}$cm^{-2}.