Porous Silicon Multilayers on Narrow Vertical Walls for Optical Applications

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Abstract

This work reports preliminary results on porous silicon multilayers formed on narrow vertical silicon walls of high aspect ratio.
Silicon walls with lengths ranging from 1 to 10mm, width from 2 to 16μm and height of 18μm were obtained using a simple and cheap chemical anisotropic etching. PS multilayers on the walls, as well as on the substrate, are formed by means of a chemical anodization process with various current densities. The structures are characterized using scanning electron microscopy, optical imaging, and Raman spectroscopy.
Raman analysis shows a multilayer structure on the walls, suggesting different refractive indices for each layer. These results indicate the possibility of obtention of visible light waveguides formed by porous silicon multilayers made on silicon vertical walls. Future investigations will focus on the improvement of superficial quality of silicon walls, as well as on the formation of PS layers with adequate refractive indices for the desired application.

1. Introduction

Porous silicon (PS) has been a material very much studied in the last years due to its photoluminescence in the visible range at room temperature [1] and its high surface-to-volume ratio [2]. It has also attracted special attention due to its possible application in the field of optoelectronic [3] and as sacrificial layers [2] to obtain microelectromechanical structures (MEMS).

The aim of this communication is to present preliminary results concerning the fabrication of PS multilayers, formed by anodic eletrochemical process on narrow silicon walls obtained by a simple anisotropic etch. PS multilayers with well-defined thickness and porosity can be used to fabricate optical structures like waveguides and interference filters, for example.

The hope of a photonic entirely based on silicon has been renewed. The condition to reach such goal is the development of light emitting diodes [4], photonic band gap devices [5], Bragg reflectors [6], all optical switching devices [7] and waveguides [8]. The use of multilayer structures formed by PS has already been suggested and demonstrated [9]. PS multilayers are possible because the porosity only depends on the current density, once the other etching parameters are kept fixed, and the refractive index of PS depends on its porosity[10].

2. Materials and methods

The first process sequence deals with the fabrication of monocrystalline silicon walls of high aspect ratio. The objective is to obtain structures with the following dimensions: width from 2 to 20μm, height from 15 to 20μm, and from 1 to 10mm in length. Moreover, it is required that they present vertical walls, as well as a good superficial definition. This task has been accomplished with the aid of anisotropic etching, as described below.

P-type silicon wafers, <100> and resistivity of 14 Ωcm were used, with a thermal oxide approximately 0,6μm thick. Photolitographic definition of oxide rectangles was performed, aligning the mask parallel to the <100>, that is, 45º rotated from the clivation lines. In the kind of process utilized here, underetching must be taken into account [11,12], since all planes parallel and perpendicular to the surface are etched in the same rate. Therefore, masks were designed in the following dimensions: 35, 40 and 45μm width combined with 1, 2.5, 5 and 10 mm length. Using these patterns, samples were submitted to anisotropic etching in KOH (7M) at a temperature of 75±2ºC.

Etch rate was determined as approximately 0,9μm/min, and samples were dropped in stirred solution for about 20min. Visual control under the microscope was utilized in order to evaluate wall size and height, besides the integrity of oxide masks.

The last step was to obtain PS multilayers on the walls. After remotion of all oxide mask on the substrate, ohmic contacts on the backside were made by evaporation of aluminium and subsequent annealing at 450°C for 30 min in nitrogen ambient. This contact is necessary to allow a homogeneous current flow in the anodization process. Anodization was performed under standard galvanostatic conditions using aqueous HF (48% wt) as electrolyte. The anodization current was supplied by HP E3631A power supplier, controlled by a computer, to form PS multilayers. Three porous layers were made applying $J=20mA/cm^2$ to obtain the lower and upper layers and $J=5mA/cm^2$ to obtain the intermediate layer. Time etch used was 7 minutes for each layer. After formation samples were rinsed with deionized H_20, ethanol and pentane to avoid capillarity forces.

The optical images and Raman spectra were obtained on the Renishaw Model 3000 Raman system equipped with a CCD detector and an Olympus BH-2 Microscope. Measurements were made in the backscattering geometry using an 80X microscope objective to focus the light source. Images were obtained by white light scattering in the sample. The Raman spectra were obtained by excitation with 633 nm Laser source of He-Ne.

3. Results and Discussion

We obtained microstructures in form of walls in width ranging from 2 to 16μm, controlling etch times, in depths of about 18μm, as it can be seen in Figures 1 and 2. A 2μm wide structure is shown in Figure 1. In this figure one can identify the loss of superficial definition of the wall and vertex, if compared to the good homogeneity of the wall from Figure 2. Because of the solution used (less than 10M), some planes are not developed, and irregularities may appear longitudinally on the corner between the wall and the base (Figure 2).

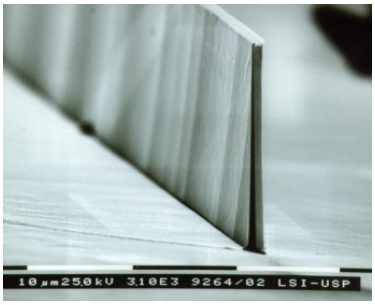

Fig. 1: SEM photography of a monocrystaline 2μm-wide vertical wall made on silicon.

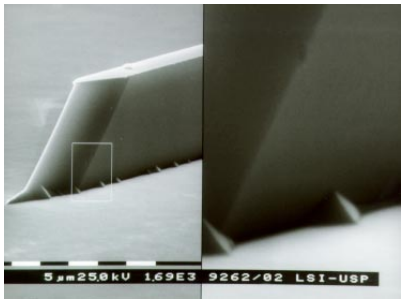

Fig. 2: SEM photography of a monocrystaline 2μm-wide vertical wall made on silicon. Amplification of the corner area.

Figure 3 and 5 show the optical images of the PS multilayers structure in the substrate and wall regions, respectively. Figures 4 and 6 show the Raman spectra obtained in the sub-regions of these structures.

The optical image of structure in the substrate showed two layers structure. The top layer was labeled with A and the internal layer was labeled as B (Figure 3). Layer A showed lower scattering intensity than layer B, indicating that layer A has lower extinction coefficient than layer B. In order to correlate with PS structure in each layer, the Raman spectra were analyzed (Figure 4). The Raman spectrum in layer A showed a peak at 504 cm^{-1} with full wide half maximum (FWHM) of 26 cm^{-1}. The Raman spectrum of layer B showed a peak at 512 cm^{-1} with FWHM of 16 cm^{-1}. These Raman lines are associated to first order LO phonon Raman line as in the c-Si (520 cm^{-1}). The peaks downshift and FHWMs enlargement of the Raman spectra in layers A and B are attributed to phonon confinement effect in the PS structures[13, 14]. Using the confinement model the average of crystallites in layer A was estimated in ca. 2.1 nm and in layer B in ca. 3nm. These results show that layer A has higher porosity than layer B, correlated with the current density during anodization process. Using the morphological parameter of PS proposed by Jaimes et al [15] and crystallite sizes it was estimated the porosity of A and B layers as ca. 70% and 57% respectively. The refractive indices of these structure were ca. 1.37 and 2.24 respectively, these values were obtained from the optical characterization of different PS layers reported by Mazzoleni et. al.[12].

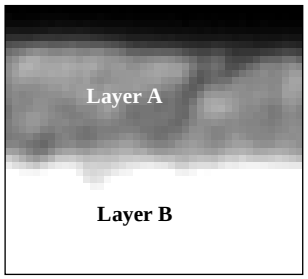

Fig. 3. Optical image of PS multilayer in the substrate region.

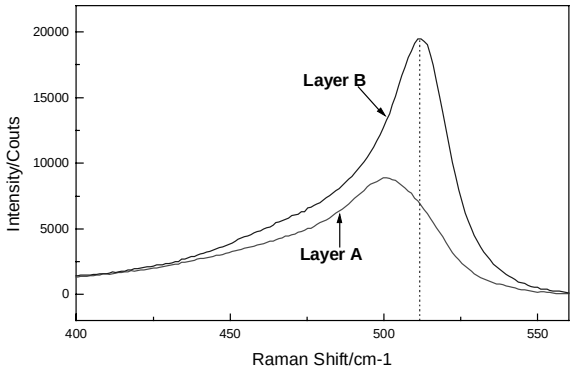

Fig. 4. Raman spectra in the sub-regions of Figure 3.

The optical image of the wall structure showed also two layers structure. The top layer and inner layer were labeled as A' and B', respectively (Figure 5). Layer A' showed higher extinction coefficient than layer B'. The Raman spectrum in layer A' (Figure 6) showed peak at the 509 cm^{-1} with FWHM ca. 17 cm^{-1}. The Raman spectrum in layer B' (Figure 6) showed peak at the 507 cm^{-1} with FWHM ca. 19 cm^{-1}. As it was described above, these Raman lines correspond to LO phonon Raman line. The average of crystallites size in the A' and B' layers were estimated in ca. 2.5 nm and 2.3 nm respectively corresponding to porosities of ca. 63% and 66% respectively. The refractive index of layer A' was ca. 1.74. Layer B' should have a little lower refractive index than layer A'.

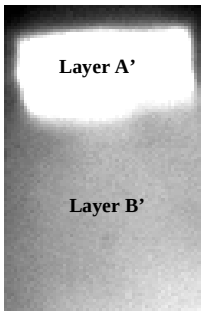

Fig. 5. Optical image of PS multilayer in the wall region.

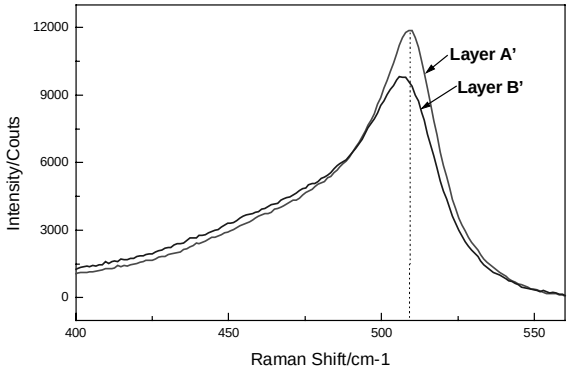

Fig. 6. Raman spectra in the sub-regions of Figure 5.

The different structural characteristic of multilayers in the substrate region relative to wall region can be explained as result of electric field nonuniform distribution in the wall during porous silicon formation but further analysis is necessary. In spite of differences of multilayer structures in the wall and in the substrate, the experimental results showed a PS layers formation in the one dimensional wall structure. These results are promising to waveguide application.

4. Conclusions

Based on the results presented in this work, the following conclusions can be drawn:

(i) Narrow vertical walls of high aspect ratio were obtained in widths ranging from 2 to 16μm with a simple and cheap chemical etching process;

(ii) PS formation in walls as a multilayer structure with different refractive index in each layer was investigated.

Our efforts are now directed to the obtention of a central layer confined between two layers with lower refractive indices in order to guide visible light.

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