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Invited Paper

Light polarization in active photonic waveguides of porous silicon

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ABSTRACT

Optical properties of porous silicon are attractive to fabricate integrated optoelectronic devices. However, the large light absorption in the visible of porous silicon and the difficulty to produce active photonic structures have limited possible applications. In this work, we combined a polyoxometalate assisted electrochemical etching with the supercritical drying process to produce an active 1D photonic waveguide of porous silicon capable of concentrating light emission. It comprises a photonic mirror cladding and a luminescent core with a broad emission spectrum centered at 635 nm at room temperature. Results showed that luminescence modes with transversal magnetic (TM) polarization are totally absorbed while transversal electric (TE) modes are guided, but it restricts the length of the waveguide to only few mm. This polarizer waveguide could find application in integrated optoelectronic devices.

1. Introduction

Porous silicon (p-Si) is a nanostructured material with a coral-like morphology produced by electrochemical etching of crystalline silicon (c-Si) in an aqueous hydrofluoric acid (HF) electrolyte. Therefore, p-Si morphology depends on c-Si electronic properties, electrolyte composition, applied electrical current, temperature, etc. The electrochemical dissolution of silicon occurs mainly at the pore tips; once a p-Si layer is formed, the walls are practically inert [1]. A quantitative estimation of this effect is given by the Beale model. First, silicon substrate must have holes, p-type doped or induced by illumination, to attract F^- ions and form a Schottky barrier in the silicon/electrolyte interface [2]. The current is transported through this barrier by electron tunneling, such that the porosity of p-Si increases with the applied electrical current density since electrons have more energy to cross the barrier. Then, the dissolution of silicon occurs mainly at the pore tips, where the electric field concentrates and decreases the Schottky barrier height. Hence new p-Si layers are produced at the etch front without affecting previous etched layers. In contrast to the low light emission of c-Si since its indirect band gap, p-Si can show luminescence in the visible region. The light emission of p-Si is characteristic of the quantum confinement of charge carriers in the nanostructure that opens the electronic bands as p-Si skeleton thickness is reduced to a few nanometers (< 4 nm, exciton Bohr radius), with participation of surfaces states [3,4]. However, an external quantum efficiency of only 10%, a wide emission band, and long recombination lifetimes have restricted optoelectronic applications of p-Si. The low quantum efficiency of p-Si could be, in part, a side effect of the collapse of the highly porous structure during sample drying that increases the number of non-radiative defects. Canham [5] has shown that the supercritical drying of high porosity p-Si powder enhances the luminescence quantum yield up to 32% at room temperature by reducing surface tension and capillary forces on the structure. Moreover, p-Si refractive index can be tuned, from approximately 1.1 to 2.7, with the electrical current applied during the electrochemical etching to produce high quality 1D photonic structures [6].

The luminescence and optical properties of p-Si are attractive to fabricate visible light sources and active photonic structures, among others. However, visible luminescent and photonic multilayers of p-Si are usually produced as separated entities, as they depend on the electrical resistivity of the c-Si substrate. Photonic multilayers produced from highly p-type doped c-Si substrates have a large refractive index contrast (around 1.3/2.7), skeleton and pore thickness around 20 to 50 nm, but show a weak luminescent restricted to the infrared. On the other hand, p-Si produced from lightly p-type doped c-Si substrates has a thin skeleton (< 4 nm), shows efficient luminescence in the visible, but this type of multilayer structures has low refractive index contrast and roughness in the layers interfaces. In this work, we used highly doped c-Si substrates to produce photonic multilayers and added a POM solution (polyoxometalate and hydrogen peroxide) to the electrolyte for luminescent layers [7]. In this way, photonic multilayers with large refractive index contrast and luminescent layers can be produced in the same c-Si substrate by exchanging the electrolyte. Furthermore, we

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combined the polyoxometalate assisted electrochemical etching of p-Si with a supercritical drying process to produce an active 1D photonic micro waveguide, with a photonic mirror as cladding and a luminescent core, both of p-Si. The purpose of this waveguide is to confine the light emission in the core plane to reduce the emission dispersion. To design a waveguide of luminescent p-Si, light absorption of p-Si in the visible had to be included in the theoretical calculations. To our knowledge, most p-Si waveguides reported in literature are passive and designed to guide in the infrared region, where the light absorption is low.

2. Experimental details

2.1. Sample preparation

The p-Si samples were produced by electrochemical etching of p-type boron doped c-Si substrates with (100) orientation and electrical resistivity of 0.01–0.02 Ω cm. An aluminum film was evaporated on one side of the c-Si wafers and heated up to 550 °C in an inert atmosphere during 15 min to make electrical contact. A Teflon cell was filled with an aqueous HF electrolyte, where the c-Si substrate was the cathode and a platinum mesh the anode. The electrochemical etching starts by applying a constant electrical current. The porosity and thickness of a p-Si layer depend on the current density and the etching time, both controlled by computer. p-Si single layers of two different porosities were produced by applying electrical current densities of 4 and 32 mA cm^{-2} (layers A and B, respectively) with an electrolyte composed of HF, ethanol, and glycerol in a volume ratio of 3:6:1 (electrolyte 1). A third layer (C) of higher porosity and luminescent was produced with an electrical current density of 79 mA cm^{-2} in an electrolyte composed of HF, ethanol, glycerol and a polyoxometalate (POM) solution in a volume ratio of 3:5:1:1 (electrolyte 2). The POM solution contained phosphomolybdic acid hydrate ($H_3PMo_{12}O_{40}$) at a concentration of 10^{-3} M in ethanol with H_2O_2, in a volume ratio of 3:1. After anodization, samples were rinsed with ethanol and kept in it before drying. Sample C was subjected to supercritical drying process in a Supercritical Fluid Technologies equipment (model SFT-100). The drying process consisted in replacing the remaining ethanol in the porous structure by CO_2 at the supercritical state to avoid capillary forces and surface tension [8]. Wet samples were transferred into a holder filled with ethanol, placed inside the chamber, and hermetically closed. Ethanol in p-Si was replaced with CO_2 at a flow of 0.2 ml min^{-1} during 7 h., at room temperature and a pressure of 10.34 MPa. Then, the chamber was heated to 40 °C at a rate of 10 °C hr^{-1}; in this step, the pressure and CO_2 flow was kept constant. Notice that pressure and temperature were slightly above the critical point of CO_2 (T_c = 31.1 °C, P_c = 7.39 MPa). Once the chamber reached 40 °C, it was vented slowly at constant temperature. Three samples of different thickness for each layer A, B and C were prepared to characterize their optical properties and the electrochemical etching rate. The porosity of each p-Si layer was calculated by gravimetry and compared to the ellipsometry analysis data. Figs. 1a-c show SEM images of the cross section of p-Si layers for different porosities.

P-Si planar waveguides composed of a Bragg mirror as cladding with a luminescent core on the top were produced by alternating the applied electrical current during electrochemical etching and electrolyte composition. First, a p-Si luminescent core (layer C) of 9313 ± 44 nm in thickness was etched with electrolyte 2 at the current density of 79 mA cm^{-2}. Below the core, a Bragg mirror of 25 periods was produced with electrolyte 1 and current densities of 4 and 32 mA cm^{-2} (A and B layers, respectively). The stop band of the Bragg mirror was centered at 753 nm for normal incidence such that the reflectance spectrum was displaced to 635 nm at grazing angle to overlap photoluminescence spectrum of layer C. After etching, p-Si waveguides were rinsed with ethanol and exposed to the supercritical drying process with CO_2 described above. Fig. 1d shows the cross section of the waveguide, where it is clearly distinguished the core and Bragg mirror. The thickness of A and B layers were 82 and 114 ± 3 nm, respectively, measured from SEM images at higher amplification than the one given in Fig. 1d.

2.2. Refractive index

The refractive index of p-Si was determined with a Wollam alpha-SE spectroscopic ellipsometer. This equipment measures the ellipsometric angles Δ and Ψ at three incidence angles: 65°, 70° and 75° in the wavelength range from 400 to 900 nm. Modeling was performed with the CompleteEASE® software supplied with the equipment to calculate the optical constants. SEM images of the cross section of the p-Si samples showed in Fig. 1 were taken as references to construct the optical models for the Spectroscopic Ellipsometry (SE) analysis. As it can be observed in Fig. 1a, the sample of lower porosity (layer A) has a coral-like structure, but as porosity increases, a column-like morphology is developed, see Fig. 1b and c corresponding to layers B and C, respectively. Furthermore, it is well known that p-Si displays a porosity gradient from the top to the bottom of the substrate that heightens in thick layers. The porosity gradient effect on the p-Si refractive index determination was averaged by measuring samples of three different thicknesses, for each porosity sample. Based on the observed p-Si morphology, we selected two models for SE analysis: gradual and anisotropic [9]. In the gradual model, the refractive index gradient of a p-Si layer is represented by a series of thin sublayers of different porosity. Each sublayer is composed of c-Si, SiO_2 and air with a slight porosity change from one sublayer to the next. This model fits well for p-Si porosity below 70%, where a coral-like structure is observed (Fig. 1a). In contrast, the anisotropic model considers the column structure developed in large porosity samples. The effective medium approach used in each model was selected according to the sample porosity, i.e. in the low porosity layers (A), the Bruggeman theory was applied, and for medium and high porosity layers (B and C) the Maxwell-Garnett theory was used. In Table 1 are shown the porosity and thickness calculated by SE analysis in comparison with the measured gravimetric data and SEM images, respectively. The porosity of layers A, B and C is around 55%, 74% and 90%, respectively. Fig. 2 summarizes the optical constants calculated from the SE analysis. In the case of layers B and C, only ordinary optical constants are shown to simplify the theoretical analysis.

2.3. Photoluminescence

Photoluminescence at room temperature of a high porosity p-Si sample (layer C) of 10 μm in thickness was measured. For this purpose, the sample was excited with a 26 ps pulsed laser of 355 nm wavelength (the third harmonic of a Nd:YAG EKSPLA laser of 10 Hz) and a fluence of 373 μJ cm^{-2}. The excitation beam stroked normally onto the sample, near one border; the photoluminescence intensity was collected from the cross section via a 10x microscope objective (Newport model M-10X) focused into an optical fiber of 1000 μm in diameter (Ocean Optics, model P1000-2-UV-VIS), and subsequently detected by a USB 2000+ Ocean Optics spectrometer. Fig. 3 shows the photoluminescence spectrum of layer C in the range from 500 to 800 nm, with its maximum emission centered at 635 nm. Layers A and B did not show any detectable luminescence in the visible region, as their skeleton thickness is larger than 20 nm. On the other hand, layer C showed visible luminescence since its skeleton thickness was reduced by the addition of the POM solution to the HF electrolyte and application of a high electrical current. The hydrogen peroxide contained in the POM solution aids to reduce the skeleton thickness by oxidizing the surface area of p-Si, and silicon oxide is further dissolved by the HF contained in the electrolyte.

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