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Synthesis and study of the optical properties of dielectric Bragg reflectors infiltrated with 6G-Rhodamine

E Gómez-Barojas[1], V Aca-López[1], E Sánchez-Mora[2], R Silva-González[2], and J A Luna-López[1]

[1]CIDS-IC, Benemérita Universidad Autónoma de Puebla, Apdo. Postal 196, Puebla, Pue. 72000. México
[2]Instituto de Física, Benemérita Universidad Autónoma de Puebla, Apdo. Postal J-48, Puebla, Pue. 72570 México

e-mail: egomez@ifuap.buap.mx

Abstract. We report the study of the optical properties of 6G-Rhodamine (Rhd) infiltrated porous silicon dielectric Bragg reflectors (DBRs) with 31 constituent periods. The DBRs were obtained by an electrochemical anodizing process of Si in a two electrodes Teflon cell. The porosity was determined by gravimetric measurements on single Porous silicon (PSi) layers. Based on the characterization results of single layers the DBRs were synthesized. After anodizing, the DBRs were silanized with a 3-mercaptopropyltrimethoxysilane solution and functionalized with Rhd solutions at different concentrations. Cross section scanning electron micrographs show that the DBRs synthesis was successful. After each preparation step, Reflectance and Fluorescence (FL) spectra were recorded. These spectra show that as the Rhd concentration in solution is increased the stop band intensity as well as the FL intensity are enhanced due to constructive interference effects.

1. Introduction

Bragg mirrors, also called distributed Bragg reflectors formed with PSi multilayers have been already suggested and demonstrated [1-3]. The most important property that the DBRs present is the high reflectivity region or photonic stop band and its wavelength range selectivity. The stop band is formed by the effect of multiple constructive interferences when a light beam is reflected by many dielectric interfaces. Infiltrating the PSi-DBRs pores with a fluorophore and tuning the DBR stop band with the fluorophore FL emission band it is possible to enhance the fluorophore FL emission intensity.
In this work we investigate the effect of infiltrating the DBRs pores with different concentrations of Rhd on the optical properties of the DBRs. These properties were studied with reflectivity- and fluorescence- spectroscopy.

2. Experimental procedure

This investigation was carried out in 2 parts. In the first part a set of single PSi layers were synthesized and characterized. In the second part, based on the experimental results obtained in the first part, a set of DBRs were synthesized by electrochemical etching. As substrate it was used polished silicon p-type wafers (1,0,0) orientation with resistivity of 0.01- 0.02 Ω·cm.
The electrochemical anodization setup is formed by an electrochemical cell and a programmable BK Precision 9130 used as a current source. The electrochemical cell consisted of: a Teflon base, a Si

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wafer (1.5 cm×1.5 cm) that acted as the anode, a neoprene O-ring of internal diameter of 0.91 cm, a small Teflon cell, and a Tungsten filament in spiral form that acted as the cathode. The etching solution contained Ethanol (purity: 99.5%), Hydrofluoric acid (45%) and Glycerol (purity: 99%). The PSi samples have been prepared by keeping the Ethanol:HF:Glycerol ratio at 60:30:10. A set of 7 PSi single layers were prepared in a Teflon two electrode electrochemical cell with anodizing current density (J) of 20, 45, 70, 95 and 120 mA/cm2 for 20 min.

The equipment used in this investigation was: Profilemeter Veeco model DEKTAK 150, an analytical balance AND HR-200, a Field Emission SEM, JEOL JSM-7800 F, a Spectrometer Varian Cary 100 Scan and a Varian Fluorescence Spectrometer Cary Eclipse model.

3. Results and discussion

The porosity of the set of PSi layers was determined by the gravimetric technique [3]. Then, a plot porosity vs anodizing current density of the set of single PSi layers was generated. The thicknesses of the PSi layers were calculated using the formula given by Vinegoni et al. [3], then, a plot of thickness data vs anodizing current density was done. With the thicknesses data of the samples, the etching rate defined as the thickness of the PSi layer divided by the anodizing process time, was determined. Then, the refraction indices of the set of PSi layers were determined using the equation given by Pap et al. [4].

The dielectric Bragg reflectors are narrow–band dielectric mirrors obtained by stacking periodically two layers of high refractive index (layer H) and low refractive index (layer L) whose thickness is λ/4. This means that the Bragg's relation $n_H d_H = n_L d_L = \lambda/4$ was used to design the DBRs where λ= 570 nm, the wavelength at which Rhd fluoresces [5] was chosen. Taking into account the experimental results obtained for the PSi single layers set, DBRs with 31 periods were fabricated. The chosen parameters are: the layer-H with n_H=1.81, P=61% and thickness of 0.78 μm. The layer-L with n_L=1.29, P=81% and thickness of 0.11 μm. Care was taken to form reflecting interfaces of H-layers with the adjoining media so that the reflecting beams interfere constructively. Due to the short anodizing process times a computer program to control the current source was used.

The as anodized (0.0 Rhd) DBRs were stabilized by immersion into a 2.5 % v/v a solution of 2-Propanol with 5% v/v of 3-Mercaptopropyltrimetoxysilane (MPTS) for 15 min. Then, they were functionalized by dipping them into a Rhd solution. The Rhd concentration was varied from 1.2 to 1.8 M in increments of 0.2 M also the 0.6 M concentration was included.

In figure 1 SEM micrographs of the surface morphology and of the cross section of an as anodized DBR formed with 31 periods by electrochemical etching are shown. The fabrication of this kind of multilayer structures is possible because the silicon anodization only occurs at the pore tips that is, at the interface between the Si substrate and the electrolyte so that the porous layer already formed is not affected by the subsequent conditions of fabrication.

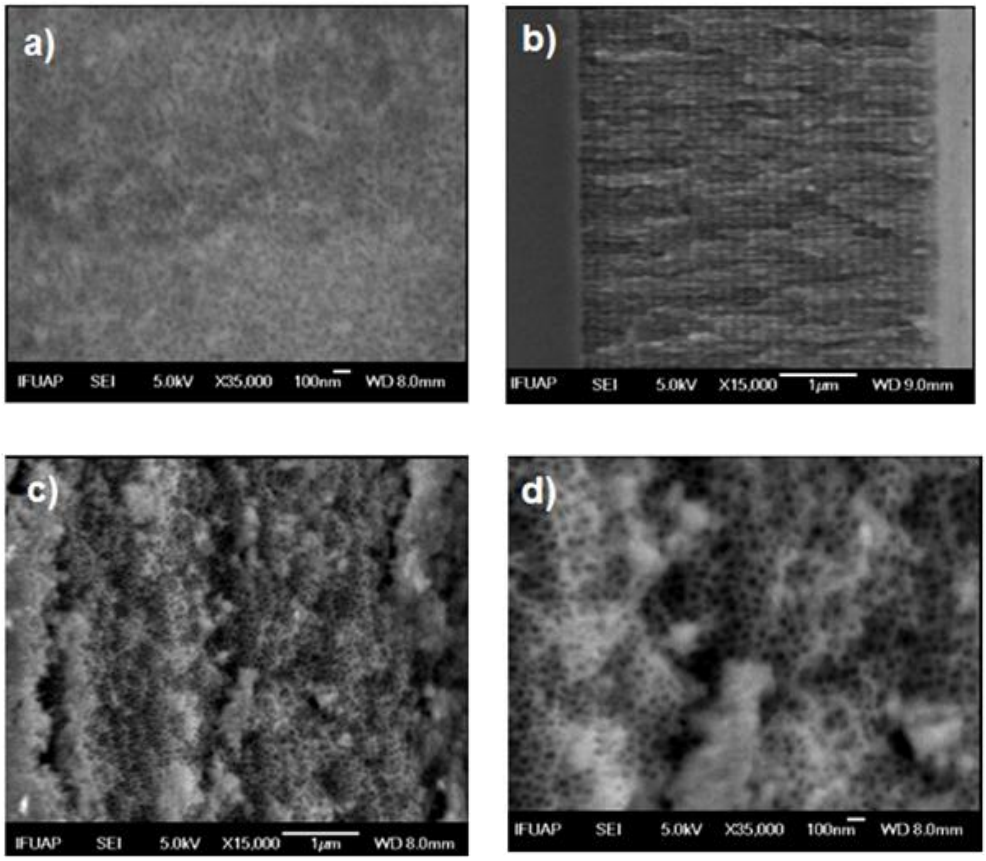


Figure 1. SEM micrographs of an as grown DBR formed with 31 periods by electrochemical etching. (a) image at x35000 of the surface morphology of the uppermost layer (b) Cross section image at x35000, it is seen clearly the constituent layers of the DBR, (c) Top down view (x15000) of a free standing piece of cross section layer of the DBR. It is seen that the fringes frontiers are not abrupt and (d) an image of the region shown in (c) at x35000 where the deep pores were focused so that they can be seen clearly in a nanometric scale.

The set of DBRs were characterized by 45º incidence reflectance measured at room temperature with an Halogen-W lamp and normalized with respect to the reflectance of an Al coated mirror. All DBRs present a stop band whose peaks are at slightly different wavelengths in the range from 478-520 nm and the amplitude of the stop bands are increased when the Rhd concentration is varied from 1.2 M to 1.8 M, see figure 2a.

Room temperature Fluorescence spectra were recorded using the λ=330 nm line of a Xe lamp as excitation source The spectrometer entrance and output slits were 10 nm wide. All DBRs present a very wide FL emission spectrum (400-800 nm) whose peaks are located at 569 nm, also it is seen that amplitude of the spectra increases as the Rhd concentration in the functionalization solution is increased, see figure 2b. The DBR functionalized with 0.6 M Rhd does not follow the tendency; the reason is that when the DBR stop band coincides with λ=570 then the FL amplitude increases due to a constructive interference otherwise a destructive interference is caused.

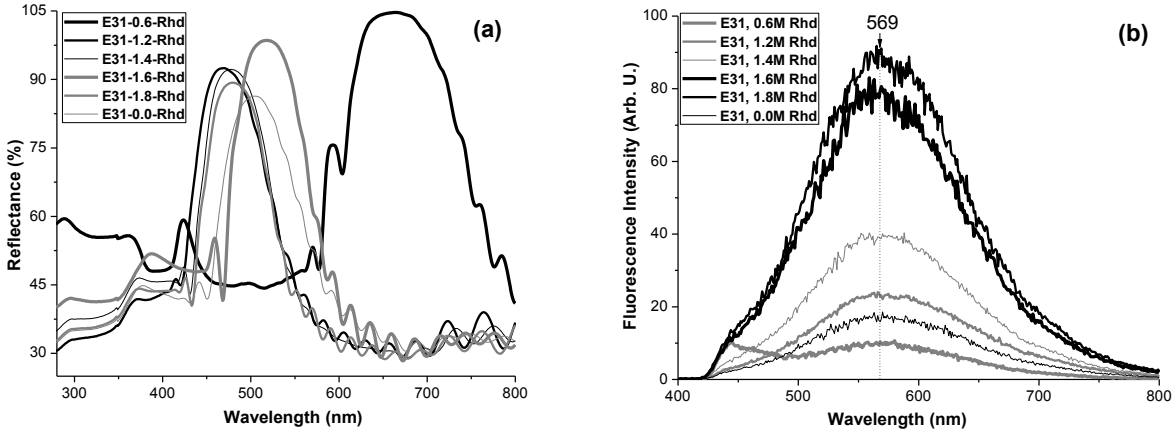


Figure 2. (a) Specular Reflectance spectra and (b) FL spectra recorded from the set of DBRs formed with 31 periods and functionalized with different concentrations of Rhd. The FL spectrum of the as anodized DBR is included for comparison.

In summary the SEM micrographs are the evidence that the BRDs were successfully synthesized by an electrochemical anodizing process, they show also interesting details of the forming layers, of their interfaces, and of the pores size. The FL spectra show that the DBRs functionalization with Rhd was successful too, since the amplitude of these spectra is increased with respect to the one of the as anodized DBR, as the Rhd concentration is increased.

4. Acknowledgments

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