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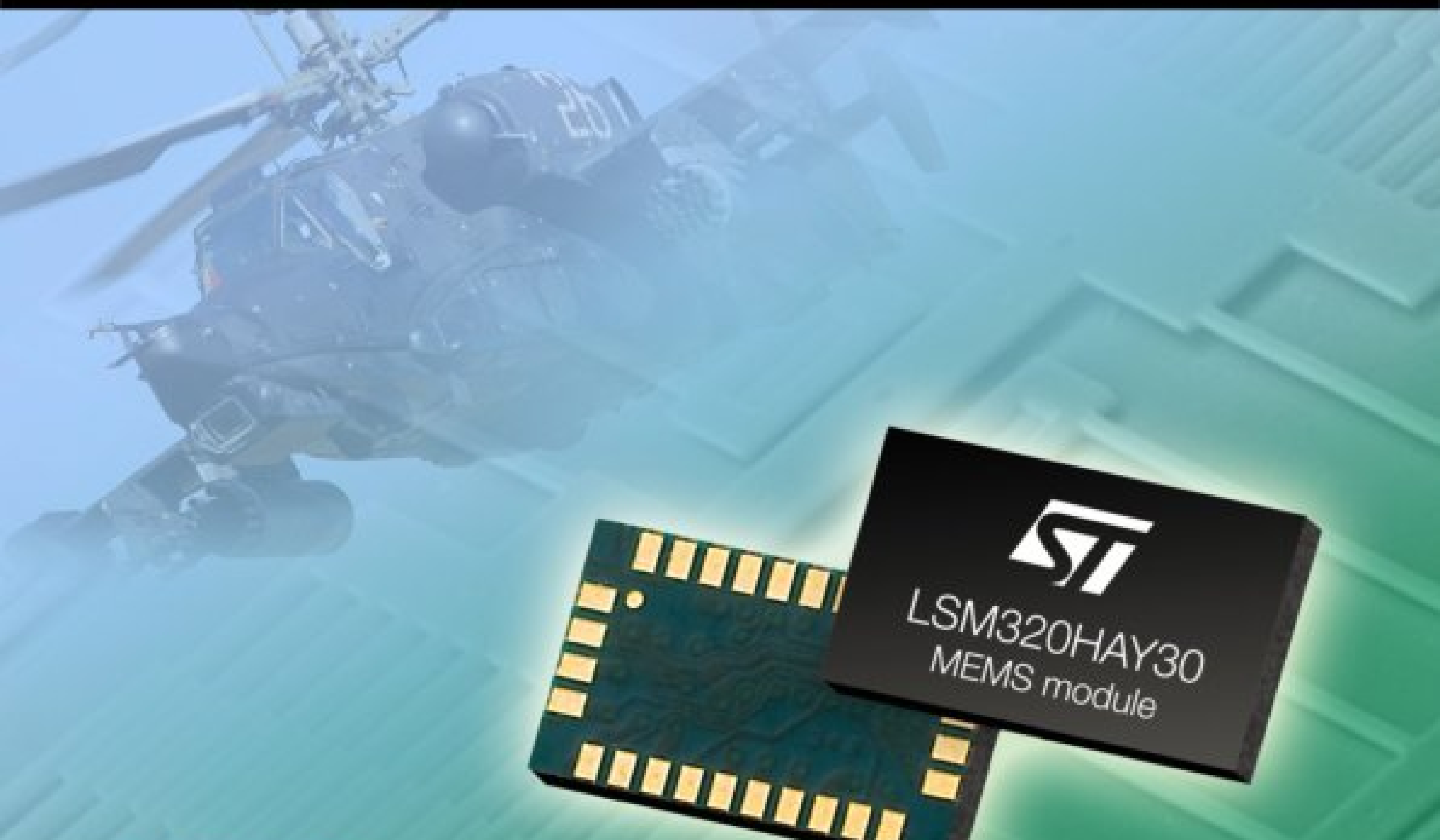

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Realization of Porous Silicon Distributed Bragg Reflector for Optical Sensing Applications

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Abstract: This paper reports the feasibility for realization of Porous Silicon (PS) based Distributed Bragg Reflector (DBR) structures for an optical sensing applications. PSDBR sensor device structure was fabricated by electrochemical anodization of crystalline silicon wafer and proposed as a large surface area matrix for optical sensing applications. The refractive index of the PSDBR structure is tuned by changing current density and the thickness by etching time. Photonic stop band wavelength in the measured reflectance spectra of sensor device structure was analyzed for the detection of the analyte in the porous structure. The sensing device performance is tested by the different organic chemicals. The photonic stop band wavelength of DBR showed the significant red shift after organic chemical adsorption because the layers refractive index was changed after chemical fill the pores of the PSDBR structure. Also the stop band wavelength shift showed a linear relationship with the refractive index of the organic chemicals. *Copyright © 2012 IFSA.*

Keywords: Porous silicon, Electrochemical anodization, Distributed Bragg reflector, Optical sensor device.

1. Introduction

PS is formed from a crystalline material, consists of nanometer to micron-sized columnar pores in a silicon matrix. Due to the unique electrical and optical characteristics, porous silicon has attracted great attention of scientists in the recent years [1, 2]. PS is an easily fabricated device that has extremely high surface area to volume ratio, making it an ideal platform for optical devices and sensing applications. DBR is the periodic dielectric structures that control the propagation of electromagnetic wave through the PS photonic crystals and exhibits the photonic stop band in the reflectance spectra. Many groups all over the world have been working in the research to develop nano-optical devices and sensors using PS for the future applications because it's optical properties are highly sensitive to the presence of chemical and biological species inside the pores [3]. Several groups have reported the applications of porous silicon nanostructures in the optical devices and sensing applications like, chemical, biochemical, solar cell, humidity, different gases, DNA biosensor and other emerging and future nanotechnology applications [4-9].

The objective of the present work is to evaluate the feasibility of realization for PSDBR structure as an optical sensor device. In section 2, detail fabrication method is discussed. In section 3, principle of optical sensing, structural and optical characterization of the PSDBR structure is discussed. In the last testing of the sensor device is done for the different organic chemicals. These results are useful for the sensing of different analytes like gas, bio-chemicals etc. also.

2. Experimental

PSDBR structure was fabricated by electrochemical etching of p-type Si wafer (<100>, 0.01- 0.02 Ohm-cm, 275 µm, 20 cm^2). The schematic diagram of the electrochemical etching cell is shown in Fig. 1.

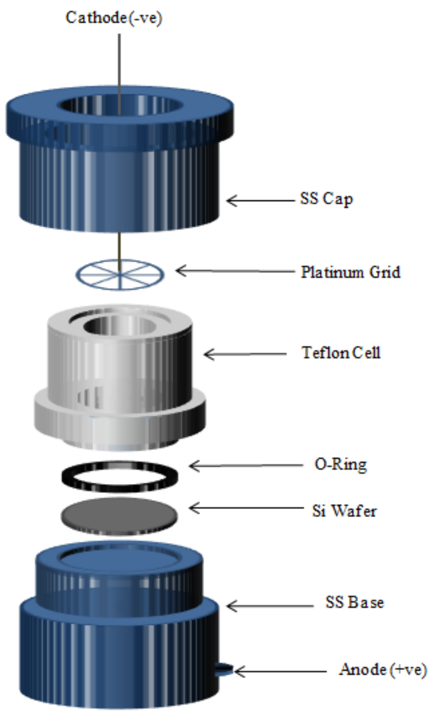

Fig. 1. Electrochemical Etching Cell.

As shown in Fig. 1, the base and the cap of the electrochemical etching cell were made with SS 320 metal. Silicon wafer was placed inside the base and sealed with an O-ring and exposed to the electrolyte. The electrolyte mixture was kept in the highly HF resistant polymer polytetrafluoroethylene (PTFE), which was in contact with the platinum grid, used as a cathode. First, silicon wafer was cleaned using standard piranha cleaning method. PTFE bath was filled with the etching solution of 40% aqueous HF and 99% ethanol, mixed in the ratio of 1:2. The cathode was immersed in the electrolyte solution and the distance between anode and cathode was kept about

4.5 cm. Periodic constant current square wave was applied by programmable DC power supply (PWS 4305, Tektronix). A constant current mode was used for anodization process as it is beneficial in terms of regulation [10]. Applied current density (*J*) and the etching time (*t*) profile are responsible for the change in refractive index (*n*) and the physical thickness (*d*) profile of the layer, respectively. The fabrication schematic diagram is shown in Fig. 2. In Fig. 2, n_s is the refractive index of the substrate and *N* is the number of periods. Multilayer PSDBR was fabricated with twenty repetitions of current density and etching time sequences as shown in Fig. 2.

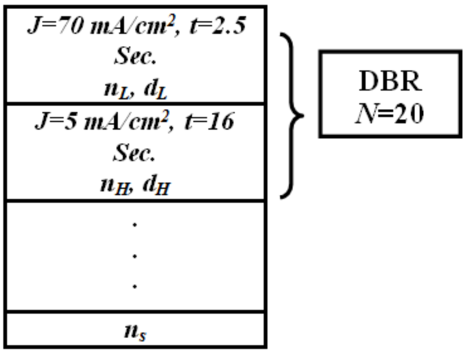

Fig. 2. Fabrication Schematic of PSDBR Sensor Device Structure.

After electrochemical etching, PSDBR structure was rinsed in DI water for 10 minutes and dried at room temperature. Structural morphology (plan and cross sectional view) was examined by SEM (CHI900B CH Instruments). The reflectance spectra were measured before, during and after exposure to various organic chemicals using spectrometer (MayaPro-2000, Ocean Optics). All measurements were done in the air. Polished silicon wafer was used as reference in the reflectance measurements.

3. Results and Discussion

According to the optics theory, the reflectance spectrum of PSDBR sensor device structure is governed by the interferometric Fabry-Perot relationship [6]. Light reflected from the top interface (air-PS) and the bottom interface (PS-Si substrate) interfere with each other and form the typical Fabry-Perot fringes in the reflectance spectrum. The fringe pattern is closely related to effective optical thickness, which is product of physical thickness and refractive index of the structure, by the relationship shown in as:

$$m\lambda = 2nd \;, \tag{1}$$

where, *m* is an integer (the spectral peak order) and *λ* is the peak wavelength. For bare single layer PS structure (without any analyte), the refractive index of the structure is *n*. When the pores are filled with an analyte (e.g., chemicals or bio-chemicals), the effective refractive index of the structure increases from n to $n+\Delta n$ with shift in wavelength from λ to $\lambda+\Delta\lambda$ in the reflectance spectra due to increased optical thickness of the structure. For the multilayer PSDBR structures with alternating high refractive index layers and low refractive index layers the Eq. (1) becomes:

$$\frac{m\,\lambda_0}{2} = n_L d_L + n_H d_H \,, \tag{2}$$

where, λ_0 is the photonic resonance wavelength, n_L and d_L are the refractive index and the thickness of the low index layer, respectively, while n_H and d_H *are* the refractive index and the thickness of the high index layer, respectively.

The prepared PSDBR sensor device structure show distinct green, blue and red colours distribution over the entire surface (Fig. 3). Porous structure in the bulk silicon is strongly responsible for the change in the surface colour due to the shifting in the bandgap energy of silicon.

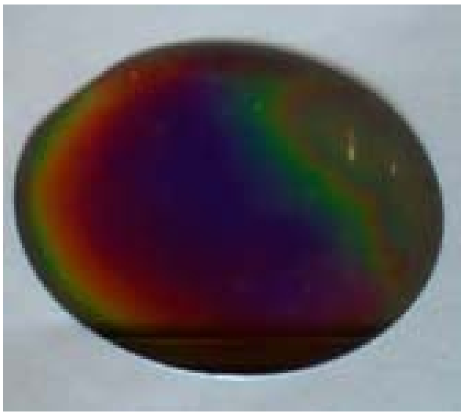

Fig. 3. Fabricated PSDBR Sensor Device Structures.

Fig. 4 shows the surface morphology of the structures in SEM plan view. The array of void spaces (dark) in silicon matrix (bright) can be seen clearly in the plan view SEM image. The morphology of the structures shows that the electrochemical etching is done uniformly on the surface and created the granular structure in a spherical shape. Large number of pores distributed in all direction can be observed in Fig. 4 with mean pore size of 24 nm measured using Image J software [11].

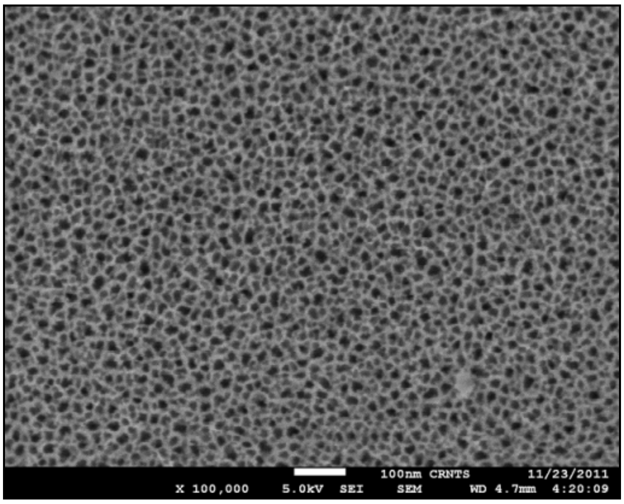

Fig. 4. SEM Plan View of PSDBR Structures.

The cross-sectional SEM image of the PSDBR structure is shown in Fig. 5. Multilayered stacks are clearly observed in this figure. These stacks are due to the periodic variation in the refractive index profile through the current density variation for different etching time. As seen in Fig. 5, the light grey stripes correspond to low porosity layers for the current density 5 mA/cm^2 (high refractive index) and the dark gray stripes correspond to high porosity layers for the current density 70 mA/cm^2 (low refractive index) . Further, the branched cylindrical structure possessed by the pores, is also clearly visible in Fig. 5; which shows that the pore growth is occurred in the depth (perpendicular to the surface) of the structure.

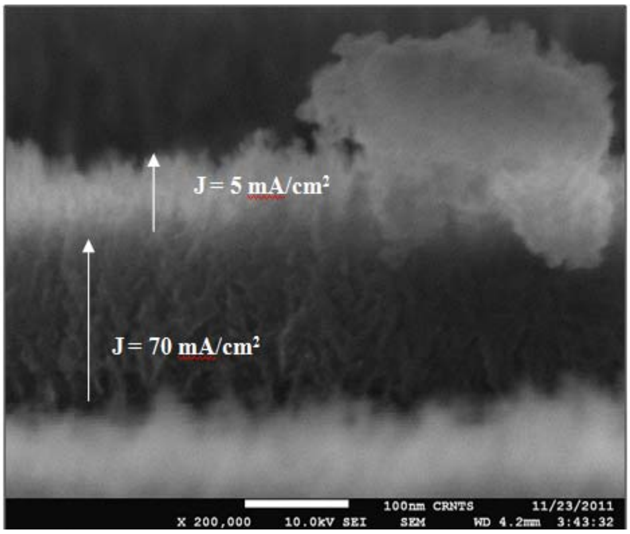

Fig. 5. SEM Cross-section Image of PSDBR.

After drying of PSDBR structure at room temperature, the reflectance spectra was measured, which is shown in Fig. 6.

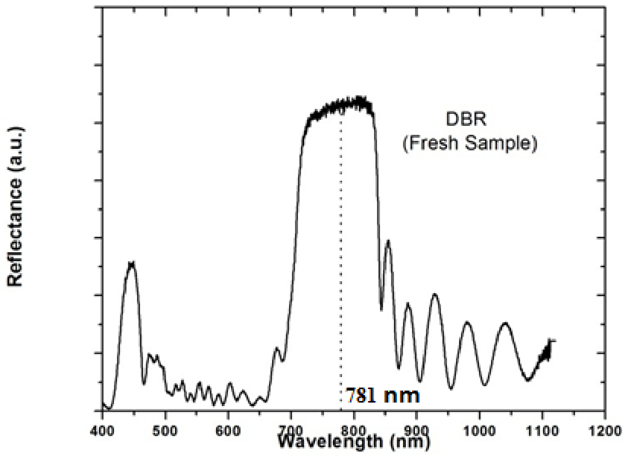

Fig. 6. Reflectance Spectra of PSDBR Structure.

The photonic resonance centered at 781 nm is observed in the reflectance spectrum of PSDBR structures. The presence of small peaks between 400 to 500 nm for PSDBR structure was observed in the reflectance spectra of Fig. 6. This is due to the refractive index dispersion at those high energies. The reason behind this might be the absorption of light by PS at that wavelength since the refractive index is not constant at that energy range [5].

After realization and characterization of PSDBR sensor device structures, their performance as the sensor device was tested by analyzing the photonic stop band shift in the reflectance spectra during their exposure to 100% concentration of different organic chemicals. Variations in the reflectance spectra of these sensing device structures during exposure to organic chemicals are shown in the Fig. 7.

During this exposure, the reflectance spectra shifted toward the low energy region. This can be explained as the substitution of the air in the pores by the organic chemicals which lead the increase of effective refractive index of layers and consequently increasing optical thickness. As the physical thickness of PSDBR structure is fixed, the shift in the photonic stop band in the reflectance spectra must be only due to changes in the average refractive index of the PSDBR due to the pores filling by organic chemicals. A red shift was observed in the reflectance spectra and stop band during exposure to every type of organic chemicals (Fig. 7). The magnitude of red shift depends on the refractive index value of the organic chemical. The variation in the photonic stop band wavelength is listed in Table 1.

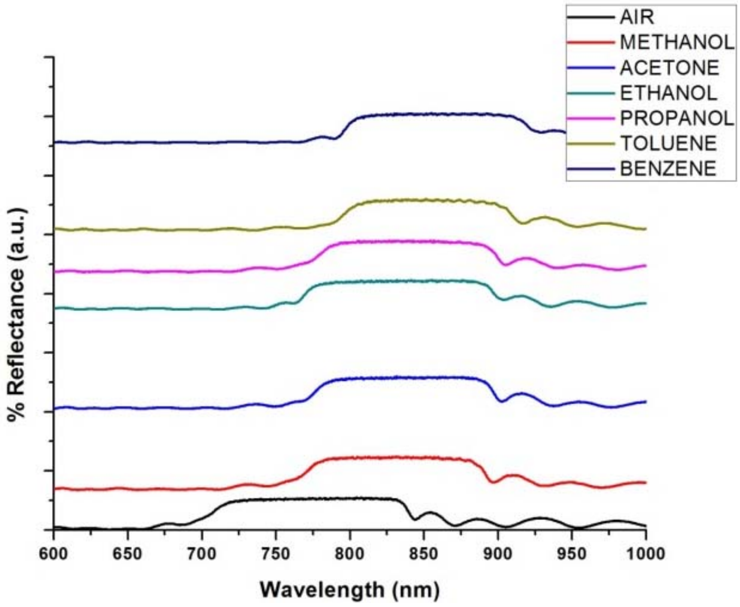

Fig. 7. The reflectance spectra of the PSDBR structure before and after organic chemical adsorption.

Table 1. The refractive index values and stop band wavelengths of the different organic chemicals.

Organic Chemical	Refractive index of Organic Chemical [12]	Stop band Wavelength (nm)
Methanol	1.331	831
Acetone	1.359	837
Ethanol	1.360	838
n-Propanol	1.385	842
Toluene	1.496	856
Benzene	1.501	860

As shown in the Table 1, when the PSDBR sensor device structure was exposed to organic chemicals of a high refractive index, large variations in the stop band wavelength in the reflectance spectra were observed; correspondingly, when the sensor structure was exposed to organic chemicals of a low refractive index, small variations in the stop band wavelength in the reflectance spectra were observed. This is due to the variations in the effective refractive index of the PSDBR layers according to the type of organic chemicals adsorbed in its pores.

Sensitivity is one of the most important issues to evaluate the performance of the sensors. In this case, the response of the sensor structure was evaluated throughout the change of the stop band wavelength in the reflectance spectrum for different chemical's refractive index which is plotted in Fig. 8 from the results of Table 1.

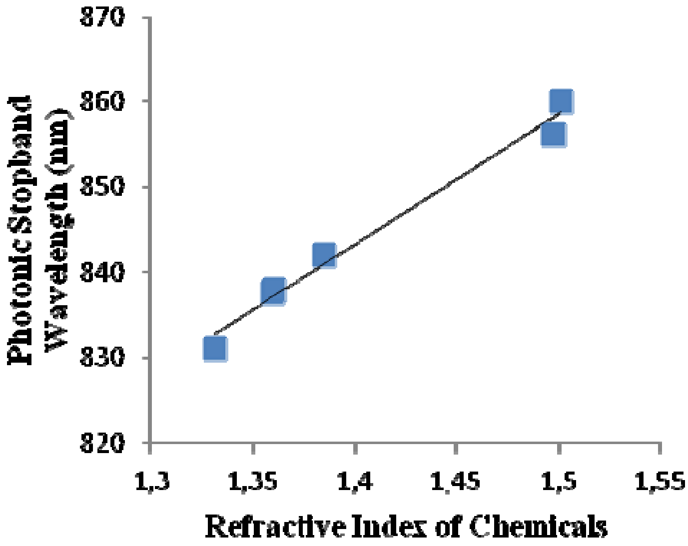

Fig. 8. The photonic stopband wavelength position of PSDBR structure.

As shown in the Fig. 8, sensor device showed to be a good linear relation ship between the refractive index of the chemical and the photonic stop band waveleength shift as a marker for sensing measurement in the PSDBR structures.

4. Conclusions

PS based DBR sensing device structure was fabricated by electrochemical etching. Porous structure is confirmed in the plan view of SEM characterization. Large number of pores uniformly distributed on overall surface is observed. Multilayered structure with periodic variation of the refractive indices is observed in the cross sectional SEM characterization. Optical characterization showed stop band wavelength centred at 781 nm. Testing of sensing device structure is done by the optical sensing of different organic chemicals. It is shown that PSDBR structure can be used as a chemical sensor for detecting different types of organic chemicals. The reflectance spectra and stopband position of this structure showed to be suitable for organic chemical detection as their photonic stop band shift is directly in relation with the refractive index value of organic chemicals. The proposed PSDBR structures can be also useful for the sensing of different analytes like gas, bio-chemicals etc...

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