Illuminations Condition Effect on Optical Properties of Nanocrystalline Porous Silicon

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Abstract
In this report, laser assisted in electrochemical etching technique PECE to prepare porous silicon layers under the constant conditions was used. To confirm the possibility of using different wavelengths of laser light to control the thickness of structures and porosity of silicon layers, a scanning electron microscope and weight measurements techniques were used to study of morphology of porous surface and its porosity. It was found that the porosity values according to the different wavelengths have same power density approximately and which ranged from 39 to 53% towards the shortest wavelength. The intensity of the integrated peak absorption of Si-H and O-H vibrations demonstrated in the FTIR spectra of these samples was calculated. In this work, optical properties dependence of porous silicon crystalline size is analyzed and correlated to photoluminescence spectra. It’s clear that the Photoluminescence PL peak energy was decreasing from 2.37 to 2.10ev, and corresponding the increasing in refractive indexes. The peak PL intensity dropped from short wavelength (blue) toward long wavelength in visible region with large than 50% ratio.

Keyword:

Porous silicon; photoluminescence ;refractive index; porosity; PECE;SEM;FTIR

Introduction

Porous silicon is remains very attractive to study because it has large photoluminescence efficiency at room temperature in the visible region(Chao Y 2018) and its one of the most important a direct band gap semiconductor material which obtained in easy and cheap techniques (R Suryana et al 2018) . It has a high absorbability and low reflectivity due to the spongy structure of porous layers (Olga Volovlikova et al 2020).To obtain a chemically stable layer (Farid A.Harraz 2014) , mechanically strong (Chiang, C., Lee, B.T. 2019) magneto-optic devices (Petra Granitzer and Klemens Rumpf 2010), anti-reflective coating (Remachea Tahmid H. et al. 2019) , waveguide (Tahmid H. et al.2019) , Chemically and biologically sensitive (Wei Li et al.2019 ; Jonathan Rassona et al. 2017) , photonic crystal (Xiaxia Yue, et al. 2019) , photodetector (Hadi, H.A. et al. 2019) and suitable for existing silicon technologies. The porous silicon layer is prepared in a number of different ways such as (Junwen Xu1 et al. 2019).metal-assisted and stain etching (M. B. de la Mora et al 2013) which are giving this possibility to many applications. The control of Porosity and thickness in order to study the optical properties of the porous silicon nanostructures open up many fields for a wide range of applications in optoelectronic technology(Arvin I.Mabilangana et al. 2016**;**YamanAfandi et al 2019;S.Praveenkumar et al2019).The photo luminescence spectroscopy allows the determination of the energy gap, refractive index and the change in the intensity beam value of semiconducting materials. Therefore, it is used to study the optical emission properties of these materials and **(**F. S. Husairi et al 2018) reported to explain the mechanism of photo emission such as quantum confinement, charge accumulation, surface distribution etc. When preparing the porous silicon layers by electrochemical methods, we note the surface of layers will be interaction with natural atmosphere and the nature of the chemical surface will be changes. Also, may change their optical properties and PL photoluminance (Laatar, F. et al 2020 ; Ramirez-Gutierrez, C.F. et al. 2019) .the current etching density , etching time , the concentration and ratio of the hydrofluoric solution, the type and orientation of the silicon substrate, the power density and type of lighting used especially in n- type were usually the most commonly used parameters to formed a nanostructure silicon. Also, others were study possibility of modifying the photoluminescence characteristics of porous silicon layers by irradiation with gamma ray (Bilenko, D.I. et al 2018) sample duration in air (Len'shin, A.S. et al 2013) and use the porosity to determines the refractive index(D. Estrada-Wiese 2018) .Many of literature were focused on density of etching current and etching time to control porosity and the thickness of the layer which will be prepared.We used PECE method to preparing porous silicon layers and we have used different wavelengths lasers in order to tunable morphology to understand the optical properties of porous silicon. The aim of this article is to study the effect of the laser wavelength was used in preparing the porous silicon layer, which plays an important role in its effect on photoluminance and refractive index of layers, as they are important for optical properties and has consideration as an antireflective feature.

Material and method

Preparation of the layers of porous silicon were using photo-electrochemical etching cell PECE as shown in fig 1. Samples of porous silicon electrochemical etching were prepared for wafers of type N mono crystalline silicon with resistance under constant conditions of current density and etching time with different laser lighting conditions during the electrochemical etching process. Firstly, the N-type silicon wafer with ordination (111) was cut into a 0.6 x 0.6 cm^2 and cleaned, respectively, with acetone, ethanol, and distilled water ultrasonically for a few minutes to remove impurities. The porous silicon layer was prepared with three different lasers (red 650nm, green 532nm, and blue 450nm) and they have same power density approximately under the following constant conditions at room temperature:

- Etching current density (J) 7 mA/cm^2,
- Etching time (T) 120 sec,
- Hydrofluoric acid concentration (HF) 40%,
- Ethanol purity more than 99%
- Volumetric ratio of 1: 1 for (HF: ethanol).

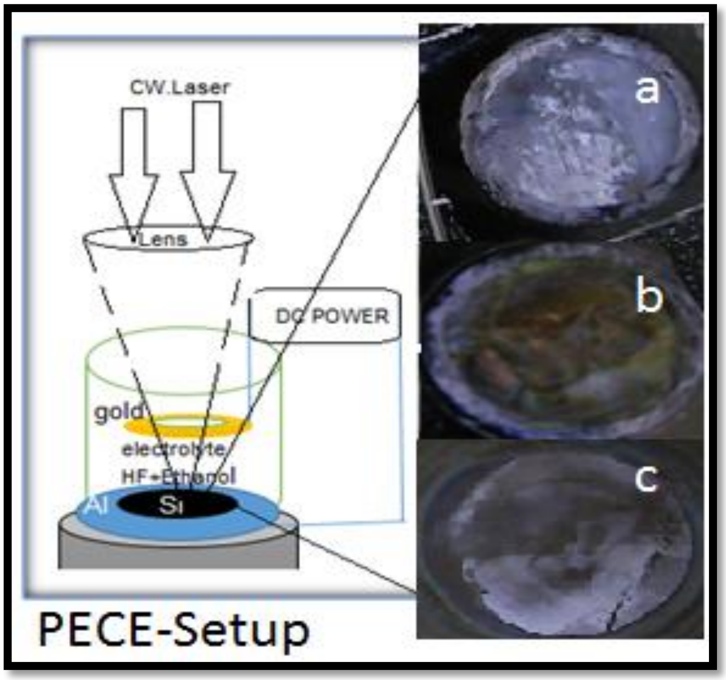

Figure 1: Schematic diagram of the electrochemical cell used for etching silicon and image of porous silicon layers with a) blue laser, b) green laser, and c) red laser

Absorption FTIR spectra are performed over range between (400- 4000)cm^{-1} in Nano Center for porous silicon layers were measured using a Shimdzu-FTIR-IR Tracer 100 spectrometer .In order to study the nanostructures of porous silicon layers morphology, scanning electron microscopy TeScan – Mira III in Sharif center for laboratory services (central Lab) Sharif University of technology was used. The PL spectra of all porous silicon samples were measured by using a spectroscopy PL 2048 TEC in Sharif center for laboratory services (central Lab) Sharif University of technology. In gravimetric method, "Mettler AE-160 digital with accuracy of 10^{-4} gm" is used to weight the samples and then to calculate the porous layer thickness and porosity.

Result and discussion

The structure and nature of the porous silicon layer is also related to the crystalline size feature of those layers and thus the change in peak-photoluminance behavior is observed specifically in the nano porous. Fig.2 displays a morphology image of porous silicon layers surface was obtained using the scanning electron microscope at different wavelengths a-blue laser ,b-green laser, and c- red laser which used in order to morphology control of porous silicon formed by electrochemical etching. It's clear that the change of wavelengths for leads to changes Pore size ranged from (59-91 nm) , (126-335 nm) and (29-51nm) for blue, green ,and red lasers respectively as shown in Fig. 2a,b,and c. As shown SEM images in Fig. 2, the highly porous layer yielded large cavities (Chiang, C., &Lee, B.T. 2019).The sizes of pores at the surface are change when illuminated the layer formed on front n-Si samples with variation wavelengths as shown in Fig.2, and the change in pore size from the surface of porous silicon to n-Si bulk is due to the transition from start pore formation to quick of pores growth.

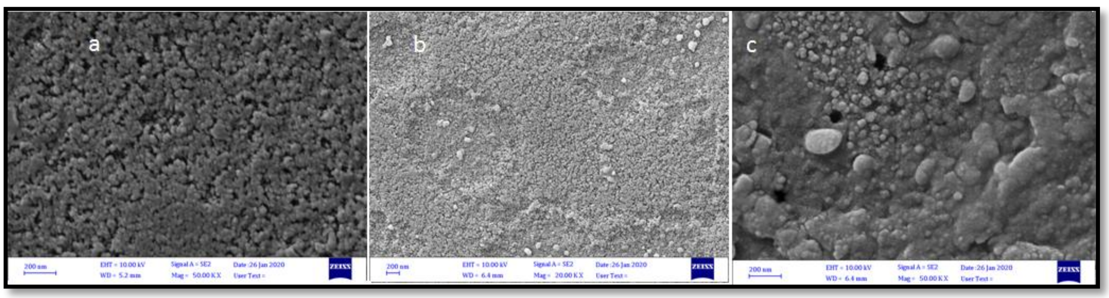

Figure 2: shows the results of scanning electron microscopy image measurement, a) blue laser, b) green laser, and c) red laser.

The porosity P% is given simply by the equation (Hasan A. Hadi 2018):

$$P\% = \frac{m_1 - m_2}{m_1 - m_3} \qquad (1)$$

where $\boldsymbol{m_1}$, and $\boldsymbol{m_2}$ are the sample weight before and after anodization, while $\boldsymbol{m_3}$ is the sample weight after removing the PS layer with KOH solution for few minutes

From these measured masses, it is also possible to determine the thickness d of the layer according to the equation (Hasan A. Hadi 2018):

$$\mathrm{d} = \frac{\mathrm{m}_1 - \mathrm{m}_2}{\rho \mathrm{A}} \qquad (2)$$

Were A is the area of the etched surface and ρ is the density of silicon.density of porous silicon can be calculated by following equation (Pavlo Lishchuk et al 2015):

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$$\rho_{PSi} = \rho_{Si}(1 - P) \qquad (3)$$

Where ρ_{PSi} is the density of porous silicon and ρ_{Si} is the density of silicon.

The porosity and thickness of the porous layer were measured by two techniques, the first one by using scanning electron microscopy for thickness layer prepared and which give indicate of effect wavelength on thickness and also gravimetric method was confirmed these effect. In the present work, a simple way was used to control of thickness and porosity of porous silicon layer. The difference in thickness and porosity which expected in the value as shown in table 1, because they have different procedure for calculates these parameters.

Table 1: The porosity, thickness and density of the porous layer were measured by gravimetric and SEM techniques for different wavelength lasers.

Samples Illumination	SEM P %	Weight P%	Density ρ_{PSi} sem	density ρ_{PSi} weight	T (µm) SEM	T(µm) weight
Red laser	39	35	1.4213	1.5145	1.2	2
Blue laser	53	56	1.0951	1.0252	1.56	0.9
Green laser	50	54	1.165	1.0718	1	1.4

Figure 3 Shows the FTIR spectra of porous silicon layers surfaces which were prepared with different laser wavelengths of (450, 532, and 650 nm) at room temperatures. When the red laser used in photo electrochemical etching PECE, the large vibration bond takes place in (611.17cm-1 wave number) as wagging mode Si – H. while, for blue and green laser used in PECE, the large vibration bonds happened centered at (611.22 cm-1) Si – H at the same mode type. For Si-O-Si bond in red laser the large vibration bond takes place in (1175 cm-1 wave number) as asymmetric stretching mode, but for blue and green laser the large vibration bonds happened centered at (1200 cm-1 wave number) as symmetric stretching (M.A. Va´squez-A. et al 2007). In the O3-Si-H and Si-H3 bonds, the large

vibration for both of them appeared at (2360 cm-1 wave number) as stretch mode for blue and green laser, while they didn't appear in red laser (Walter Jaimes et al 1997).

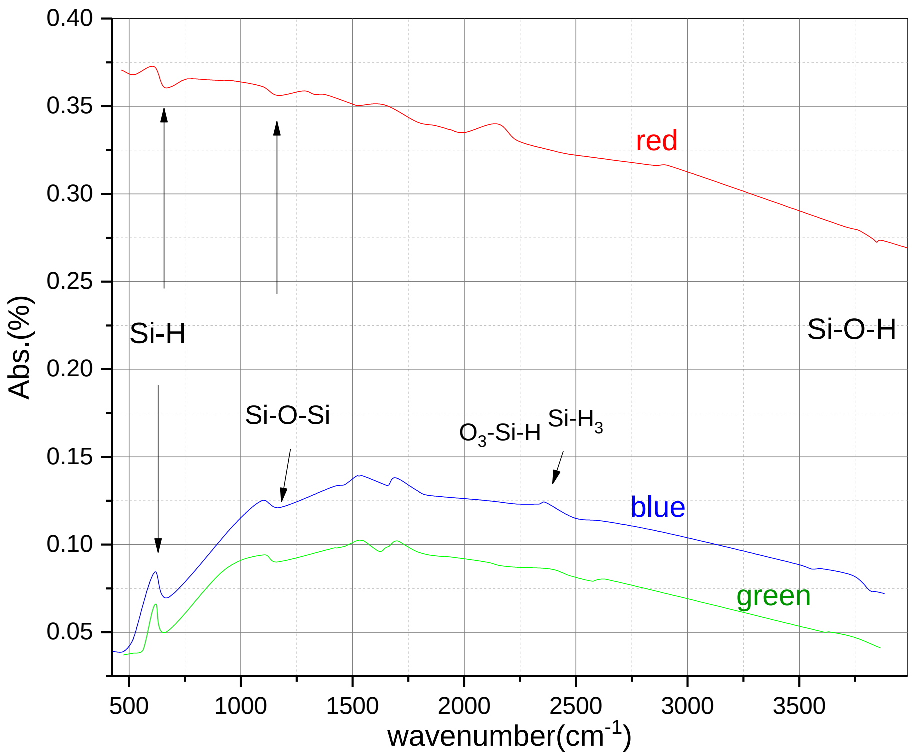

Figure 3: FTIR absorption spectrum of porous silicon layers from500 to 4000 cm^{-1} for different wavelength of red laser, blue laser, and green laser

Figures 4,5 and 6) show the photoluminescence spectra of the nanostructures porous silicon layers prepared by electrochemical etching with three different illumination condition (blue laser, green laser and red laser) respectively at room temperature and approximately the same power density. Also, at clear (see table 2) that the blue shift in visible region from 2.01 eV gap band (at red laser used) to 2.37 eV band gap (at blue laser used) because of nanostructure of the porous silicon layer which resulting quantum confinement effect of the nano crystal porous silicon layer and also the increased happen in the concentration of oxygen atoms in the formed pores and the substitution of hydrogen atoms by the oxygen atoms.

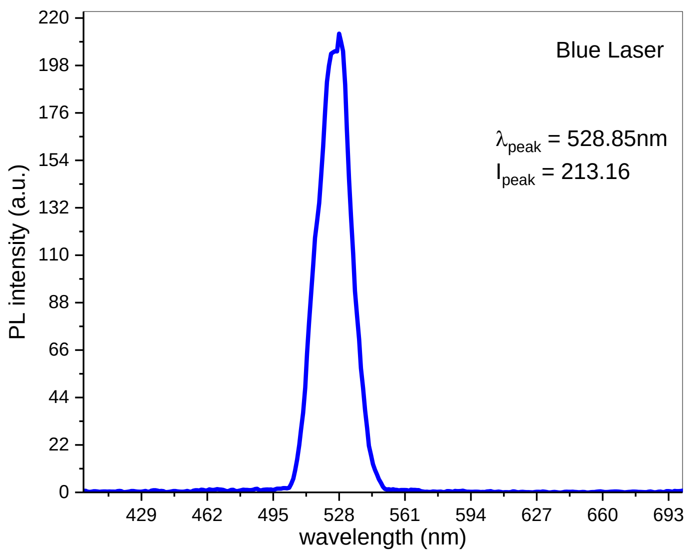

Figure 4: the photoluminance spectrum recorded under the influence of illumination blue laser

From Figures 4, 5 and 6, it is clear that the relative intensity of peaks increases about twice (213-99.59) when the wavelength decreases from red to blue in the visible region. The peaks at wavelengths 528, 588, and 592 nm are most likely due to the direct (intrinsic) transitions, so may be the transition due to the interface effect of impurity or defects (extrinsic) is less than when using high-energy light effects.

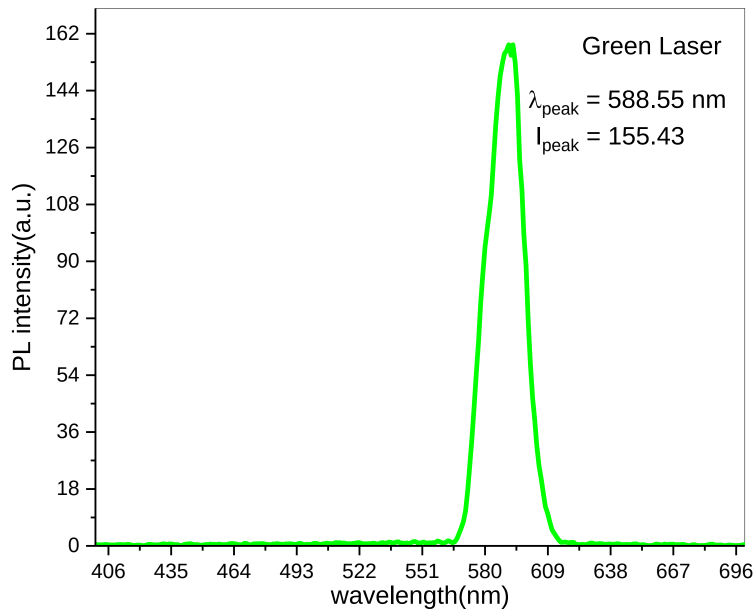

Figure 5: the photoluminance spectrum recorded under the influence of illumination green laser

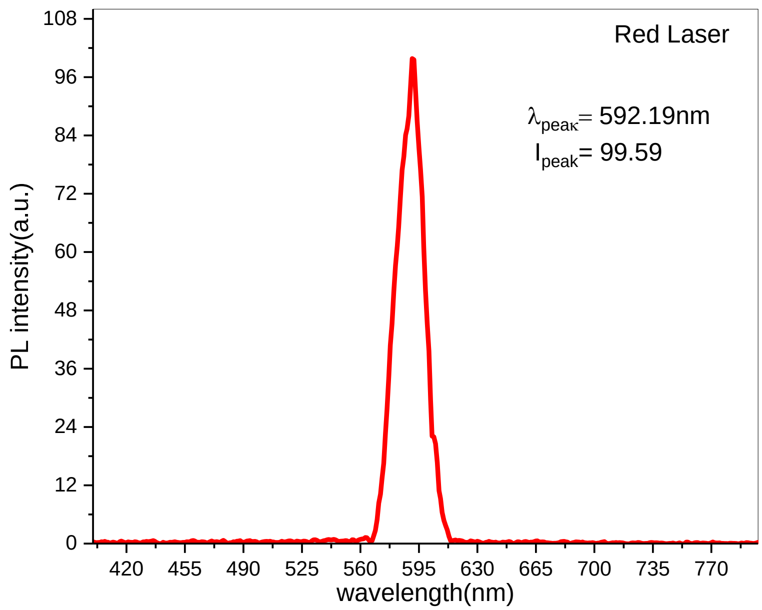

Figure 6: the photoluminance spectrum recorded under the influence of illumination red laser

Table 2: The peak intensity, peak wavelength, and band gap energy of porous silicon samples calculated by PL spectra at different wavelengths laser used

Illumination type (nm)	PL – peak Intensity	PL- peak wavelength	PL- peak energies E_{Psi}(ev)	Nano-crystal diameters (L).nm
Blue laser	213	528	2.37	2.19
Green laser	155	588	2.11	2.58
Red laser	99	592	2.10	2.59

It was observed that the porosity, thickness, and photoluminance are modifiable through illumination conditions, which makes change the peak energy gap, refractive index, and the optical properties of porous silicon also modifiable.

According to the (S. Praveenkumar et al 2018) and by using the various mathematical relationships between refractive and energy gap has been used (D. Bellet & G. Dolino,1996

;D.K. Ghosh et al 1984 ;P.J.L. Hervé& L.K.J.1995) the refractive index n_1, n_2,and n_3 can be determined using following equations:

$$n_1 = a + bE_g \qquad (4)$$

where a and b are constant and it is given as 4.048 and -0.62 per eV respectively.

$$n_2 = \sqrt{1 + \left(\frac{a}{E_g + b}\right)^2} \qquad (5)$$

where a and b are constant and it is given as 13.6 eV and 3.4eV respectively

$$n_3 = \sqrt{1 + \frac{a}{E_g + b}} \qquad (6)$$

Where a and b can be modified as a= 25Eg + 212, b = 0.21Eg +4.25

Refractive index and energy gap data were calculated and listed as shown in table 3.The refractive index value of the porous silicon layers were decreases with increasing of wavelengths toward red laser in visible region which was used to formation layers of porous silicon and enhance the rate of absorption, hence used for applications with high absorption.

Table 3: refractive index calculated according energy gap of PL for three modules (D. Bellet & G. Dolino,1996 ;D.K. Ghosh et al 1984 ;P.J.L. Hervé& L.K.J.1995)

Illumination type (nm)	**PL- peak energies E_{Psi}(ev)**	**n_1**	**n_2**	**n_3**	**Nano-crystal diameters (L).nm**
Blue laser	2.37	2.537	2.559	2.520	2.19
Green laser	2.11	2.735	2.660	2.590	2.58
Red laser	2.10	2.741	2.664	2.593	2.59

The PL of porous silicon layers reduces with the increase of wavelength. Fig.6a, b and c show the image profile of photoluminescence spectra samples for blue, green and red laser respectively. The peak photoluminescence intensity dropped from short wavelength (blue) toward long wavelength in visible region with large than 50% ratio (from 213 to 99) as shown in table 1.

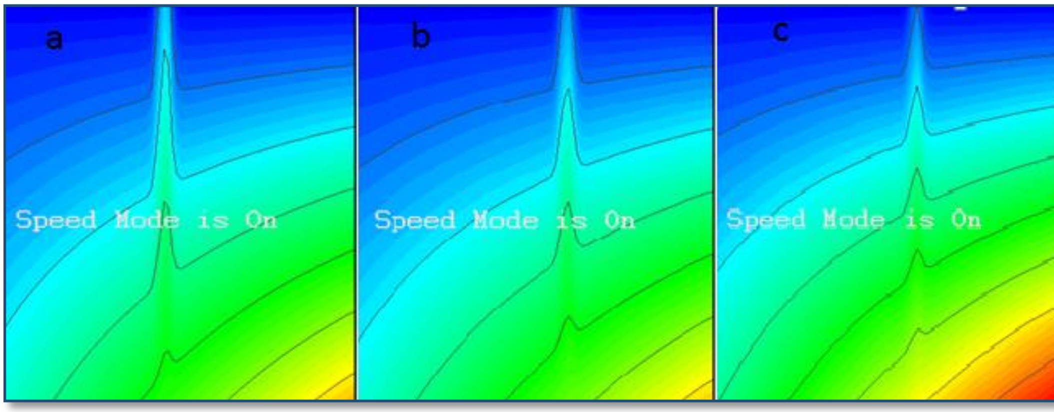

Figure 7: Image profiles of PL spectra porous silicon prepared with a) blue laser) green laser, and c) red laser

Conclusion

The photoluminescence in nano porous silicon layers can be explained in terms of nano crystalline of porous layers quantum confinement, and also due to oxides or hydrogenated surfaces of porous silicon layer, therefore we can interpreter why photo luminance spectra PL peak shifted toward to blue shift of samples prepared under illuminated at blue laser wavelength.

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