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# The Effect of Some Experimental Parameters on the Properties of Porous Silicon

*The influence of halogen lamp illumination intensity and HF acid concentrations on the properties of n-type porous silicon samples during the light-induced etching process were investigated. The photoluminescence (PL) spectra were recorded for porous silicon samples prepared at high illumination intensity. The peak and the shape of PL spectra are function to illumination intensities. The etching rates and porosities increases with increasing light beam intensity and go through maximum with increasing HF acid concentration.*

**Keywords:** Photo-chemical etching, Porous silicon, Photoluminescence  
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## 1. Introduction

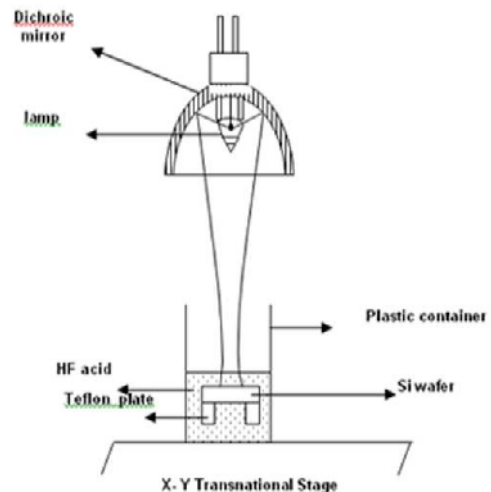
Technological application of porous silicon (PS) as a light emitter would have a significant impact on numerous technologies such as display panels or integrated circuits with optoelectronic devices on board. Such devices require strong luminescence intensity. Porous silicon consists of a network of nanometer – sized silicon regions surrounded by void space [1]. In order to obtain PS with desired dimensions and working quantities, electrochemical etching was employed [2-4].

The formation of porous silicon in HF acid with out external biasing (Photochemical etching process) was first reported by Noguchi and Suenumu by using a photon such as a high power density laser to supply the required holes in the irradiated area of the silicon wafer to initiate the etching [5]. The illumination wavelength and type of illumination (single wavelength or broad spectra) of the samples during or after the photochemical etching process is known to be an important etching parameter which can be used to modify the morphology and the photoluminescence spectra of the PS [6-8]. The purpose of this work is to prepare a PS and study the role of the halogen lamp illumination intensity and HF acid concentrations on etching rate, porosity and photoluminescence properties of (PS).

## 2. Experiment

Fig.1 shows a schematic diagram of experimental set-up for photochemical etching process. A commercially available mirror-like n-type (111) oriented wafer of (4.3-5.6) $\Omega$ .cm resistivity was rinsed with acetone and ethanol to

remove dust and with dilute (HF) to remove the native oxide and then immersed in electronic grade HF acid. The immersed wafer was mounted on two Teflon plates and irradiated at normal incidence on the polished side in a such away that the current could pass from bottom surface to light irradiation area on the top polished surface through the electrolyte as shown in Fig. (1).



**Fig. (1) Experimental set-up for Light-induced etching**

In this electrode less photochemical etching process, there was no applied bias. The light beam of quartz Tungsten halogen lamp integral with dichroic ellipsoidal mirror has been focused on a silicon wafer to a circular spot (0.75cm<sup>2</sup>) area, the distance between the halogen lamp and the wafer about (4cm). Bubbles were observed during the etching process. Wafers were etched

for 10 min at different illumination power and different HF acid concentration  $I_1=66.7\text{W/cm}^2$ ,  $I_2=133.3\text{W/cm}^2$ ,  $I_3=200\text{W/cm}^2$  and  $I_4=333.3\text{W/cm}^2$  and 10%, 20%, 30% and 40% respectively, after which they were rinsed with ethanol and dried in stream of nitrogen gas. The porous layer was formed on the mirror-like side of wafer. The value of the mean porosity over the thickness of PS layer and the thickness of PS layer were determined gravimetrically. Photoluminescence measurement was done by using He-Cd laser at a wavelength of 325nm with a low laser power density of nearly  $10\text{mW/cm}^2$  in the School of Physics, Nanostructures and Optoelectronics Research Center (NOR Lab) at the University Sian in Malaysia.

### 3. Results and Discussion

Fig. (2) shows the relationship between the light beam intensity and the etching rate. The rates were estimated from dividing the porous layer thickness by the etching times. We can easily distinguish two regions: one at intensity up to  $200\text{W/cm}^2$  there is a relatively large increase in the etching rate, while the second at higher intensities, the etching rate tends to saturate (level up). The change in the slope indicates most probably a change in the reaction mechanism [9].

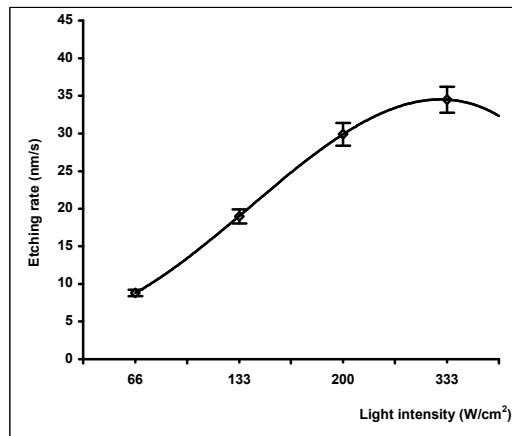


Fig. (2) the effect of light beam intensity on the etching rate of silicon nanocrystallites (nc-si),  $\rho=(4.3-5.6)\Omega.\text{cm}$

This increasing in the etching rate is due to the photon absorption of the ordinary etching light with it, larger band width from near infrared region to visible where the peak of the lamp emission around ( $\lambda=0.9\mu\text{m}$ ) and this will provide a good absorption due band to band transition and also due the absorption by the impurities especially at the larger wavelengths, the absorption length which is depended on the wave length of the light is varied from (sub  $\mu\text{m}$  to few  $\mu\text{m}$ ) [10], this will lead to hole formation at a

different depth from the polished surface and depletion layer will form in the silicon wafer such that the polished surface is relatively positive while the back side of the wafer is negative. This created a net flow of charges from the negative to the positive side resulting in a current flow which will be completed by ions flowing in the HF solution. This net perpendicular flow of charges across the wafer will encourage the etching rate in the direction of the illumination [2, 11].

Figure (3), shows that, at a given intensity, the etching rate goes through a maximum as the HF acid concentration is increased, the maximum occurring at an HF acid mass concentration of around 30%. As the concentration of HF acid increases, there are more electron accepters in the solution, resulting in an increased rate of charge transfer between the semiconductor surface and the electrolyte [9]. Thus, band bending is larger at higher HF acid concentrations. For highly concentrated solutions of HF acid, more than 30%wt, band bending is reduced by formation of ion complexes in the solution [9, 11].

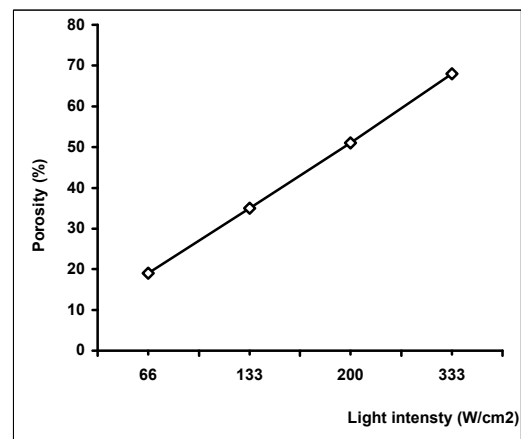


Fig. (3) The effect acid concentration on nanocrystallites (nc-si),  $\rho=(4.3-5.6)\Omega.\text{cm}$

Figure (4) shows the relationship between the light beam intensity and the porosity of the resulting porous silicon. The increasing in the illumination intensity is associated by increasing the porosity of the porous layer in approximately a linear relation this behavior is related to the silicon dissolution in the porous layer with increasing intensity [12].

Figure (5), illustrates the photoluminescence spectra of the porous silicon samples, the spectra were recorded for samples illuminated by different light beam intensity ( $I$ ) 50, 100, 150, 250 and 30% HF acid concentration. The PL spectra were not obvious at low pumping intensity while are clear at high rate  $I_2$  to  $I_4$ . These behaviors reflect the nature of the porous layer especially the morphological case.

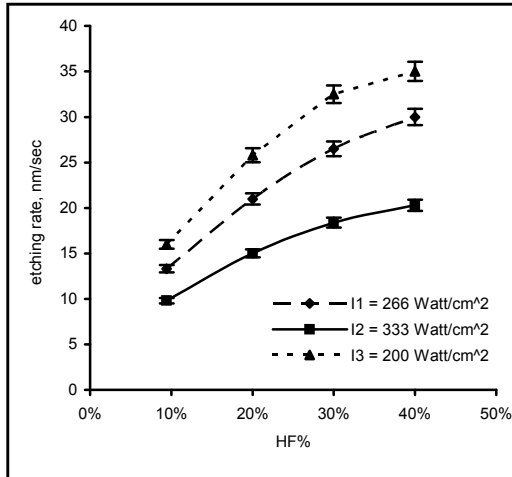


Fig. (4) The effect of light beam intensity on the porosity of PSi

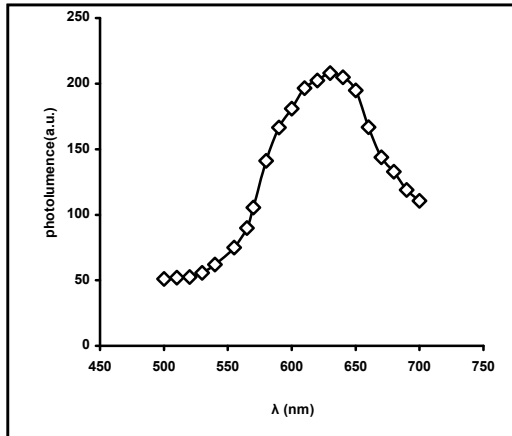


Fig. (5) PL for PS Prepared under 100W and 30% HF concentration

At low illumination intensity the porous layer may contain disconnected pits and these pits are commenced with the initiation of etching process on silicon wafers, i.e. the samples has low porosities, i.e., large size of nanocrystallites. Increasing the illumination intensity will increase the number of these pits leading to form a porous structure, [11] this structure was clear at the samples with high porosity. The increasing the porosity in porous silicon with  $I_2$  illumination samples lead to form the nano-scale silicon [1,13-15]. In this type of silicon the photoluminescence properties at room temperature is recorded by many authors [1,8,15]. The peak of PL prepared in illumination in Fig. (6) is about (360eV) while at  $I_3$  illumination intensity is blue shifting to (229eV). This is attributed to size of silicon nanocrystallites, according to quantum confinement (QC) effects [15], and

$$E_{g_{new}} = E_{g_{bulk}} + \frac{88.34}{L^{1.37}} \quad (1)$$

the average nano-size is (2.006eV) for Fig. (6) and (2eV) for Fig. (5).

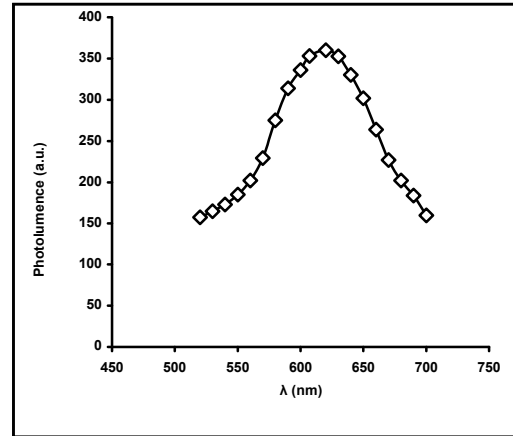


Fig. (6) PL for PS Prepared under 150W and 30% HF concentration

As shown in Fig. (7), the PL properties for samples shows in addition to the blue shifting, presses a new peak at (2.1eV) in addition to the (3.03eV) peak. This behavior refers to form a double porous layer one at low nano-size about (410nm) and another at (580nm). This may due to the excessive etching of the first layer leading to form small nano-size. This layer may presses at the surface of PS layer [12,15].

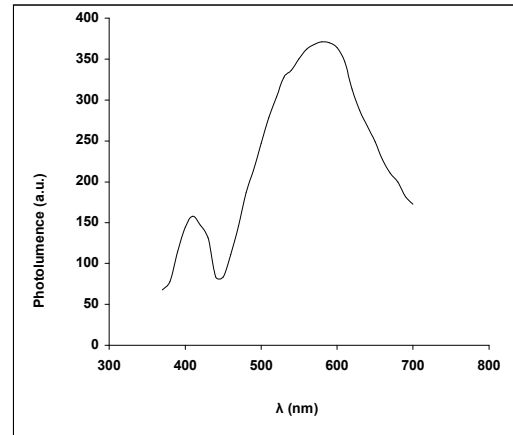


Fig. (7) PL for PS Prepared under 250W and 30% HF concentration

#### 4. Conclusion

The variation of the HF acid concentration and illumination intensity during porous silicon formation causes dramatic changes in the porous silicon properties. We found that the etching rates and the porosity of the porous layer increases with illumination intensity of the halogen lamp and go through maximum with increasing HF acid concentration. The main reason for increasing the etching rate and hence the porosity might be related to electron hole generation by the light radiation and transport holes to the surface. The PL spectra is observed just from the porous silicon samples at high illumination intensities only. The PL spectra this

may relate to the fact that at high porosities the amount is a function to the illumination intensity with a blue shifting.

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