

## Study the Properties of Silicon Nanocrystallites Prepared By Wet Etching

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### Abstract

This work presents the formation of porous silicon by photo-electrochemical processes using diode laser 514 nm, 2mW, under different etching times. The time dependence of porosity values, layer thickness, pore diameter, pore shape, wall thickness, and etching rate were studied based on SEM images.

### دراسة خصائص بلورات السليكون النانوية المحضرة باستخدام التنميش الرطب

#### الخلاصة

هذا العمل يتضمن تكوين او تصنيع السليكون المسامي باستخدام الطريقة الكهروكيميائية المحنته بالاضافة الى استخدام ثنائي ليزري طوله الموجي 514 نانومتر وطاقته 2 ملي واط تحت تاثير ازمان تفاعل مختلفة. تم دراسة اعتمادية المسامية على زمن التفاعل وسمك طبقة المسامة المتكونة كما وتم حساب قطر المسامة كما تم دراسة شكل المسامة وسمك جدار المسامة ومعدل التنميش هذه الدراسة تمت بالاعتماد على صور SEM

**Keywords:** Porous Silicon, Laser beam, SEMImages.

### Introduction

The strong visible light emission in porous silicon (porous Si) [1,2] has received a great deal of attention in last decade, owing to its scientific importance in the application of optoelectronic materials [3].

The photosynthesized PS layer produced by laser etching has a circular shape with a diameter either close to the laser beam diameter or less than the beam diameter depending on the various laser processing

Parameters like laser power density, irradiation time, and the laser

Wavelength [4]. Laser-Assisted Wet Etching (LAWE) has been investigated for microelectronics fabrication. LAWE offers some possible advantages, such as, processing at relatively low temperatures, and beam-controlled anisotropy [5]. The structural characteristics of porous silicon have been studied by a range of techniques. Direct images of the material can be carried out by scanning electron microscopy [6].

*Lehman and Gösele* [7] reported the diameter of porous silicon layer (PSL) pores or channels and found to range from 1 up to 100 nm associated with porosities of (20-80%).

In this work, we report the influence of irradiation time of laser illumination on the structural properties of n-type PSi samples during the etching process.

### Experiment

n-type Crystalline Silicon wafer with resistivity of 10  $\Omega$ .cm, 508  $\mu$ m thickness, and (100) orientation was used as starting substrates. The substrates were cut into rectangles with areas of 1 cm<sup>2</sup>. The native oxide was cleaned in a mixture of HF and H<sub>2</sub>O (1:2). After chemical treatment, 0.1  $\mu$ m-thick Al layers were deposited, by using an evaporation method, on the backside of the wafer. Photo-electrochemical etching was then performed in a mixture 48% (1:1) HF-Ethanol at room temperature by using a Pt electrode. Current of 30 mA/cm<sup>2</sup> was applied; samples were illuminated by (514 nm, 30mW) diode laser with different etching times from 1 to 15 minutes. The etched area of sample was 0.6 cm<sup>2</sup> for both cases. The structural properties: porosity, surface morphology, layer thickness, pore diameter, wall thickness, pore shape and etching rate were measured. These measurements were achieved by depending on scanning electron microscopy (SEM) images (Leo-1550). These images were performed in Institute Fuer Biologic-and Nano-systems (IBN2), Germany.

### Results And Discussion

For clarifying these sketched data in figures. (1,3) which are plotted according to SEM images, we believe that the simple one-

dimensional model suggested by *Choy and Cheah* [8], is acceptable for our cases. We chose only three SEM images for three samples, so as

clear the role of irradiation time on porous silicon formation.

Figure (2a,b, and c), depicts schematically the etching process; figure (2a) represents the Gaussian profile of a laser beam, while figure (2b) shows the profile of an etched spot on the Si sample, and figure (2c) represents the microscopic structure inside the etched area. At the beginning of the etching process, the wafer is etched in three directions; two spreading out along the surface from the center of an etched site, and one into the Si wafer, and the corresponding rates are designated as  $V_x$ ,  $V_y$  and  $V_z$ , respectively. The etching rate  $V_x$  is assumed to be the same as  $V_y$  and less vigorous than that of  $V_z$ . We attributed this behavior to the intensity profile of the Gaussian beam; the etching rate at the center of an irradiating area is higher than the others because it received the most intense light. Since the sample is an n-type silicon wafer, excess holes will be generated on the irradiated surface, thus a depletion layer will form in the silicon wafer such that the irradiation surface is relatively positive with respect to the other side of the wafer. This creates a net flow of electron from the back side to the irradiated side, resulting in a current flow through a circuit which is completed by ions in the HF aqueous solution. This net perpendicular flow of charges across the wafer will encourage the etching in the direction  $V_z$ . Based on the above analysis, we assume that  $V_z$  is more vigorous than  $V_x$  and  $V_y$ . After a certain etching

time,  $t > 0$ , a porous silicon layer is formed [fig. 2(c)] with thickness  $D$ , which is thinner than the absorption depth of the incident laser. It is reasonable to assume that at this stage  $D$  is narrow enough as shown in Fig. (3), to have an even distribution of charge carriers in the direction of  $V_z$ , so  $V_z$  will remain constant with respect to depth. However, the lateral etching rate  $V_x$  and  $V_y$  will slow down in comparison with initial rate as they move away from the center of the Gaussian laser beam. In fact, the etching rates will decrease continuously from the center of the laser beam to its periphery. According to *Noguchi* [9], the etched surface was found to be proportional to the Gaussian profile of a laser beam. For a longer etching time,  $t \gg 0$ , the porous silicon layer thickness  $D$  is further increased and the concentration of the charge carriers changes. To time more than 10 minute, the formed Si columns would become too long, for the incident light to reach the base of the silicon columns. Then, the downward etching would be reduced to almost zero. In this case, sideways etching at the surface would become dominant and result in a gradual erosion of the columns. As the columns shorten sufficiently, light can reach the lower part of the Si columns again, therefore, increasing electron-hole pair generation. At this stage, there are two etching paths the process can follow: (1) etching of the silicon substrate restarts again, and columns are formed; (2) all etching sites are passivated by hydrogen. This means fluorine cannot react with Si-Si backbone. Thus the etching stops. From the above results in figures (4 and 5), and SEM

images, we can arrive at several facts:

1. The porosity value of PSi layer depends on etching time.
2. The pore diameter depend on laser-irradiation time.
3. The surface of PSi layer was rough and its varied coarseness is in accordance to etching time.

We explain these results as follows: for longer etching time, there would be a larger etched surface to emit light as shown in Fig. (6). Apart from this, the porous layer will also expand into the bulk; the irradiated region will, therefore, enlarge. Therefore, the porosity will increase, Fig. (7) shows that the etching was extremely vigorous. The center region seemed to be removed; large holes and pores with large pore diameter are formed. These holes were linked together with some fine grooves and gaps. From Figure (4) we can note that the emitted light and porosity values from etched samples varied from sample to other. We attributed this result to the dependence of the emitted light on porosity value. (SEM) photos, taken from these samples give further evidence on the above discussion. From figure (7), nevertheless the pore number is less than that in figures (6 and 8), but its etched area is larger than these in those figures. Therefore the porosity value is higher than these in other samples. On the other hand, the number of pores in figure (8) is greater than that in figure (6), This confirms the described result in Fig. (4), for these two samples. These results are supported by [9\_12].

**Conclusions**

From the obtained results, increasing the etching time will lead to changing the morphological properties and porosities of n-type P<sub>Si</sub> to high values. The etching rates, layer thickness and porosity have maximum values at an etching time 10 min.

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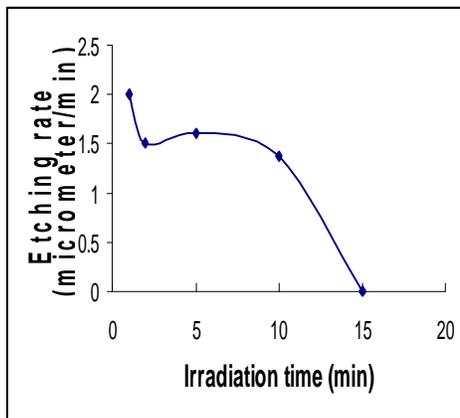


Figure (1) The relation between the etching rate and laser-irradiation time

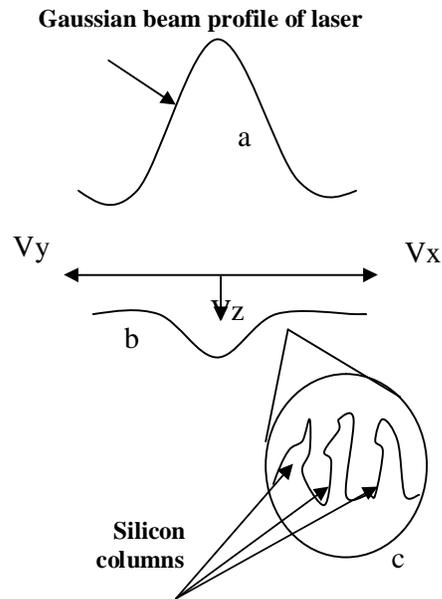


Figure. (2) A schematic diagram depicts the etching process.

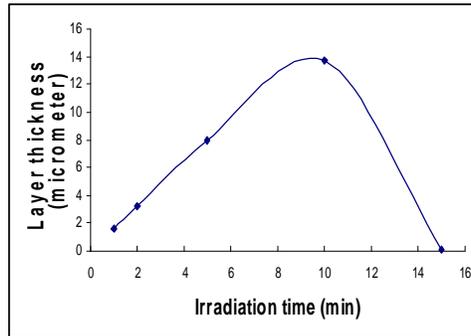


Figure (3) The relation between Layer thickness and irradiation time.

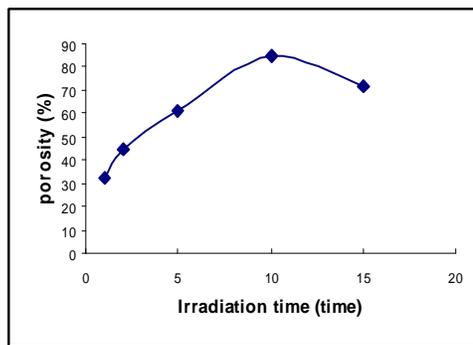


Figure (4) Effect of Laser-irradiation time on porosity

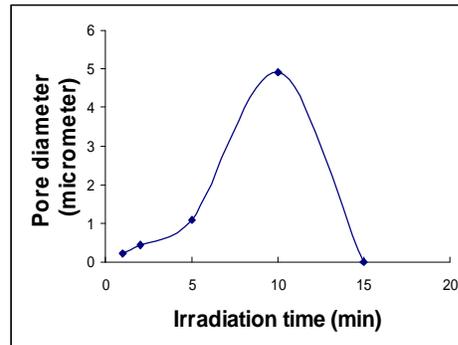


Figure (5) Effect of Laser-irradiation time on pore diameter

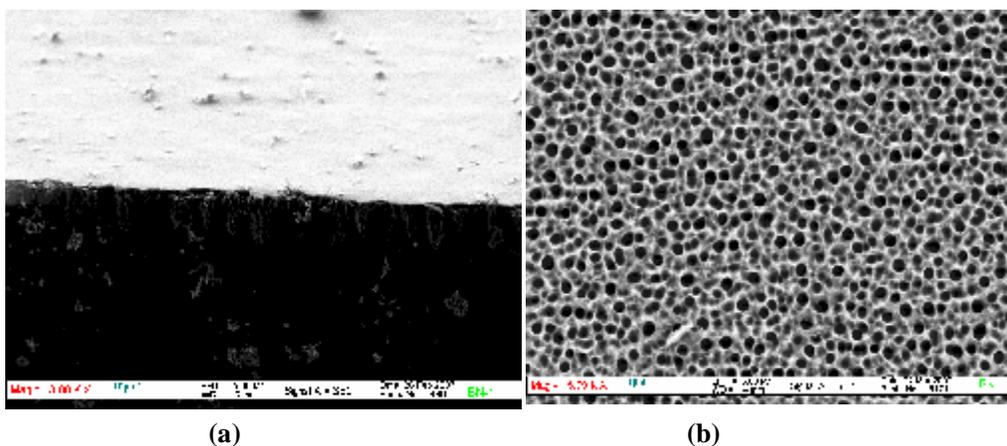


Figure (6) SEM Image of formed porous silicon layer with Laser radiation at 5 min, 30mA/cm<sup>2</sup>, 1:1HF-Ethanol. a- edge-view, b- top-view.

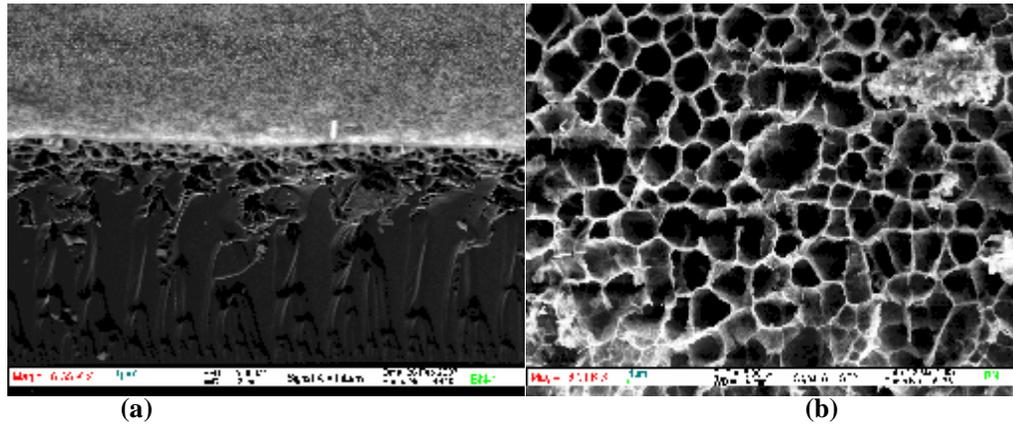


Figure (7) SEM Image of formed porous silicon layer with Laser radiation at 10 min,  $30\text{mA}/\text{cm}^2$ , 1:1HF-Ethanol. a- edge-view, b- top-view.

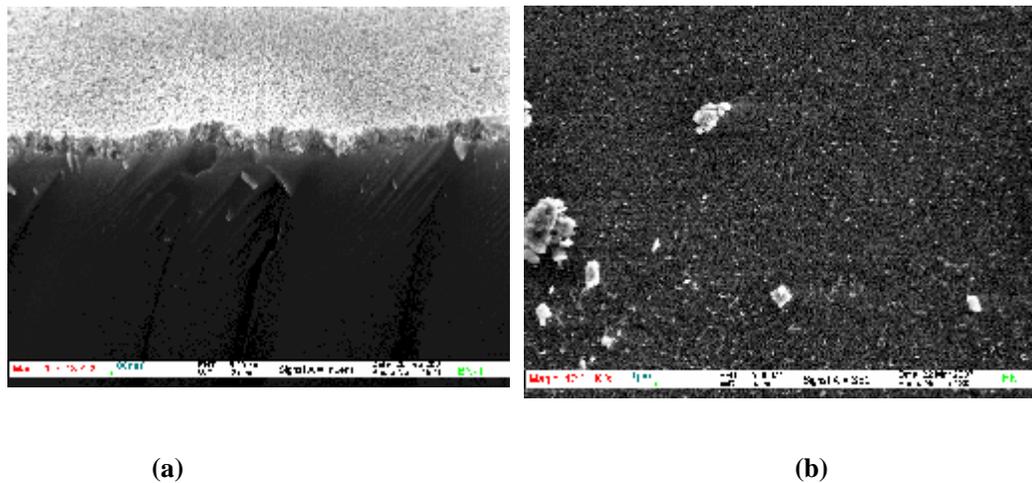


Figure (8) SEM Image of formed porous silicon layer with Laser radiation at 15 min,  $30\text{mA}/\text{cm}^2$ , 1:1HF-Ethanol. a- edge-view, b- top-view.