

## Preparation of porous silicon Wafers using sun light photo chemical etching (SLPCE)

I. K. Jassim<sup>1</sup>, A. Y. Khudair<sup>1</sup>, H. Asker k<sup>2</sup>

<sup>1</sup>Department of Physics, College of Education for Pure Science, Tikrit University, Tikrit, Iraq

<sup>2</sup>Directorate of Education Kirkuk, Ministry of Education, Kirkuk, Iraq

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**Corresponding Author:**

Name: A. Y. Khudair

E-mail: [avas47778@gmail.com](mailto:avas47778@gmail.com)

Tel:

### Abstract

Porous Silicon (PS) Layers were prepared by photochemical etching using Sun Light as a Source of energy for the first time instead of industrial Sources of light Such as Lasers, Halogen and tungsten lamps. That was used to preparation (PS) by photochemical etching. Silicon Wafers n-type (100), resistivity (10Ω.cm), (40%) HF Acid, with different illumination intensities(power density) (1758, 3956, 6182 and 8902)mw /cm<sup>2</sup> were obtained using different Lenses with different diameters (40, 60, 75 and 90) mm, focal length (13,18,41,45) cm respectively and constant etching time (60min). The morphology of the surface was studied by using an (atomic force microscope) (AFM) and (scanning electron microscope) (SEM) results shows formation of groups of pores and crystals in (nm)size with different diminution are distributed in etched area in wavy forms. The thickness of porous layers ranged from (50.06 3.68, 3.79 and 3.39) nm and the diameter of the particles are(84.06, 59.42, 51.12 and 34.8)nm respectively. The current voltage characteristics for AL<sub>thin</sub>/n-si/PS/AL<sub>thin</sub> device fabricated by sun light photochemical etching technique showed a rectifying behavior which improved with increasing light intensity.

### Introduction

Crystalline silicon is the main material for microelectronic technology but its application is limited due to its relatively small and indirect band gap of approximately (1.12 eV)[1]. In 1990 Canham found strong visible room temperature photoluminescence emitted from porous silicon (PS) [2]. an explosion of studies has been initiated to develop silicon based electroluminescent devices from this material commonly referred to as porous silicon [3]. porous silicon (PS) is a complex network of pores separated by thin columns and contains nano-meter sized Si crystallites [4]. (PS) is quickly becoming an increasingly important and versatile electronic material in today's fabrication technology. Reducing dimensionality of bulk silicon to nano-scale silicon (PS) leads to appreciable changes in optical, electrical and electronic properties. Porous silicon as suggested by Canham, Lehman and Gosele consists of a network of nanometer-sized silicon region surrounded by void space[5].

The energy band gap of nano scale silicon ( $E_{confi}$ ) is larger than that of the crystalline silicon due to the quantum confinement It is given as:

$$E_{confi} = E_{bulk} + \frac{\hbar^2 \pi^2}{2} \left[ \frac{1}{L_x^2} + \frac{1}{L_y^2} + \frac{1}{L_z^2} \right] \left[ \frac{1}{m_e^*} + \frac{1}{m_v^*} \right]$$

where  $E_{bulk}$  is energy band-gap of crystalline silicon,  $m_e^*$  and  $m_v^*$  are the effective masses of conduction and valence bands, respectively,  $L_x$ ,  $L_y$  and  $L_z$  are the dimensions of confined region[8]. The energy band gap of the nano-scale silicon as a function to the crystallite diameter according to the effective mass theory as shown in Figure (1).

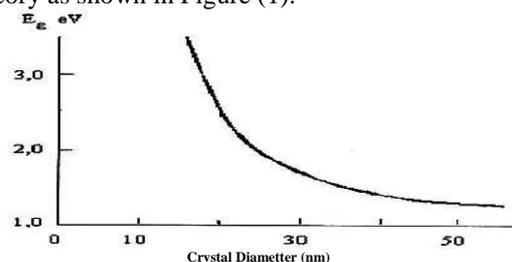


Fig (1) The band gap energy ( $E_g$ ) as a function of the crystal diameter [10].

In this research, a new and innovative method was used to prepare porous silicon for the first time suggested by (Dr. H. Asker.K 2010)[6]. Including the use of the sun as a light source in photo chemical etching method. Porous silicon can exhibit a large variety of morphologies and particle size. It has been reported that the quantum confinement effect [7]. PS are widely used in solar cell application. The porous silicon could be used in laser applications. It is used as an optically excited PS embedded in  $\text{SiO}_2$  matrix [1].

### Experimental Procedure

Crystalline silicon commercially available n-type (Si) with resistivity of  $10\ \Omega\cdot\text{cm}$  and (100) orientation were employed as substrates with dimensions of  $1\ \text{cm} \times 0.5\ \text{cm}$  to prepare (PS) by photo chemical etching. The sun is used as a light source in this research, a method used for the first time, by using lenses with different diameters (40,60,75,90)mm, focal length [13,18,41,45]cm respectively, to focus the sun light and obtain different light intensities (power density) (1758, 3956, 6182, 8902) $\text{mw}/\text{cm}^2$  respectively. Si samples were rinsed with acetone and ethanol to remove dirt followed by etching in dilute (10%) HF acid to remove the native oxide layer. The silicon samples after cleaning were immersed in electronic grade (40%) HF acid. The immersed samples were mounted on two Teflon Plates (U-Shape) as shown in fig(2) with suitable slope making the plate facing sun light with constant etching time (60min). Bubbles were observed during the etching process, indicating the propagation of the etching process and hence the formation of porous silicon. The morphology of silicon nano crystallites studied by the atomic force microscope (AFM) and scanning electron microscope (SEM). Also we studied (I-V) characteristic to measure the electrical properties and other related properties of the silicon nano crystallites Layer after etching. An ohmic contact for the prepared samples are produced from both sides (bulk & porous) by evaporating Aluminum (AL). Silver past

used to connect the copper wires on the surface of the samples.

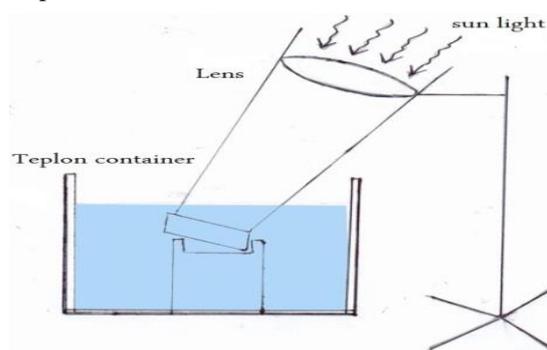


Fig (2) a schematic the Diagram of the sun light photochemical etching (SLPCE)

### Result and Discussion

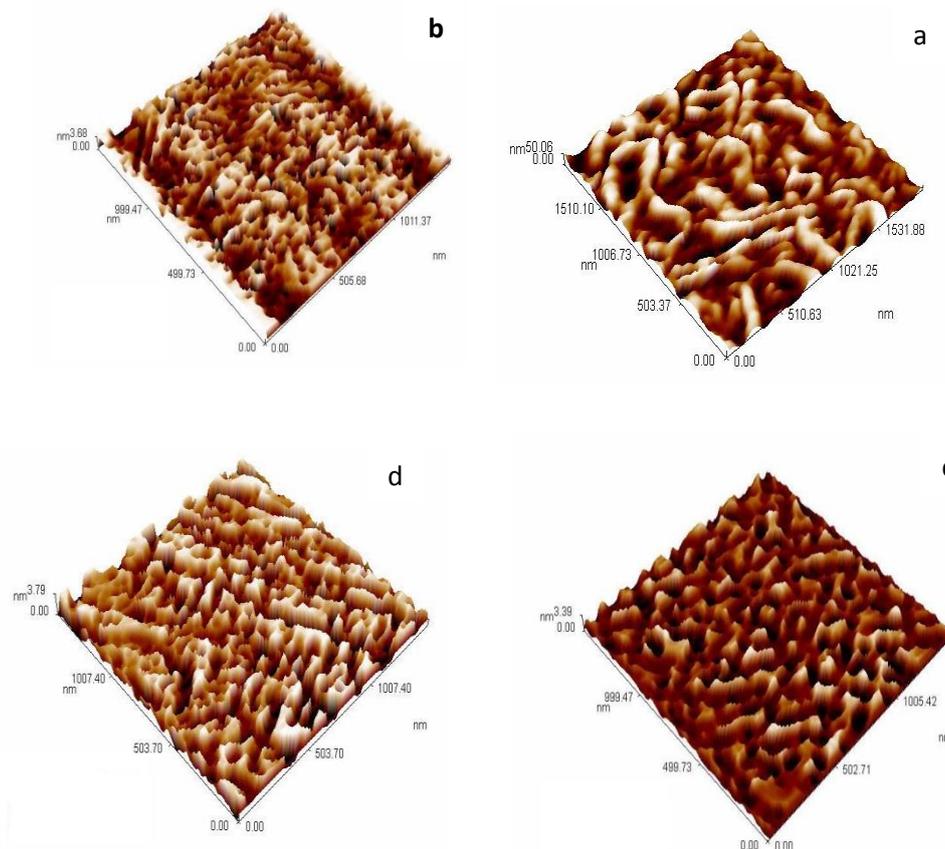
#### a- The surface morphology [AFM]

An atomic force microscope (AFM) was used to study the surface morphology of the porous silicon produced by sun light photochemical etching.

(AFM) Images for surface morphology showed the effect of light intensity on surface porosity, roughness, particle size that can be calculated and gives the distribution ratio of the crystalline particle size on the surface as shown in fig (3).

To study the effect of the light intensity on morphology of the (PS) layer, four samples of (100) n-type silicon,  $10\ \Omega\cdot\text{cm}$  resistivity immersed in (40%) HF acid for 60 min constant etching time and different light intensities (1758, 3956, 6182 and 8902)  $\text{mw}/\text{cm}^2$  focus on mirror like side of silicon samples.

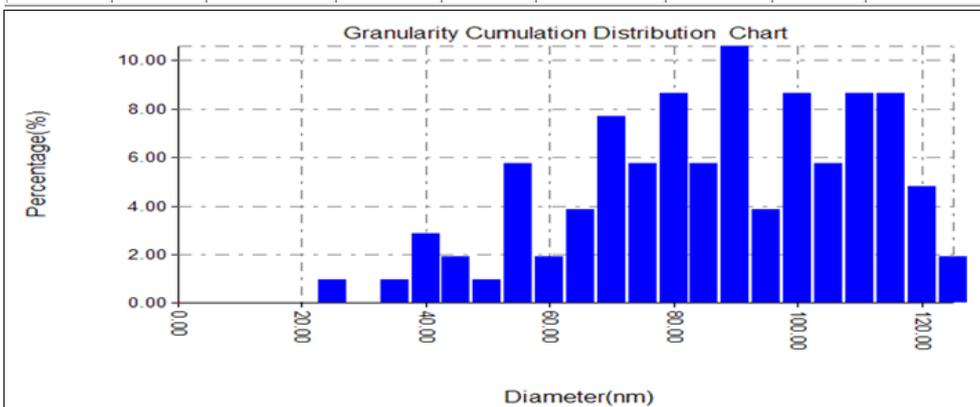
AFM studies show that the thickness of porous layers ranged from (50.06, 3.68, 3.79 and 3.39) nm, and the diameter of the particles are (84.06, 59.42, 51.12 and 34.8) nm respectively. We observe that the nano crystal size reduced with increasing light intensity.



**Fig (3) AFM Image for surface morphologies (100) n-type Si samples immersed in (40%) HF acid for constant etching time (60min ) prepared by sun light photochemical etching with different power densities:  
(a)1758 ,( b) 3956 , (c) 6182 , (d) 8902mw /cm<sup>2</sup>**

Avg. Diameter:84.37 nm	<=10% Diameter:50.00 nm
<=50% Diameter:85.00 nm	<=90% Diameter:110.00 nm

Diameter (nm)<	Volume (%)	Cumulation (%)	Diameter (nm)<	Volume (%)	Cumulation (%)	Diameter (nm)<	Volume (%)	Cumulation (%)
25.00	0.96	0.96	65.00	3.85	19.23	100.00	8.65	70.19
35.00	0.96	1.92	70.00	7.69	26.92	105.00	5.77	75.96
40.00	2.88	4.81	75.00	5.77	32.69	110.00	8.65	84.62
45.00	1.92	6.73	80.00	8.65	41.35	115.00	8.65	93.27
50.00	0.96	7.69	85.00	5.77	47.12	120.00	4.81	98.08
55.00	5.77	13.46	90.00	10.58	57.69	125.00	1.92	100.00
60.00	1.92	15.38	95.00	3.85	61.54			



**Fig (4) Distribution of statistical size nano particles made up on surface of the sample (a) in fig .(3)**

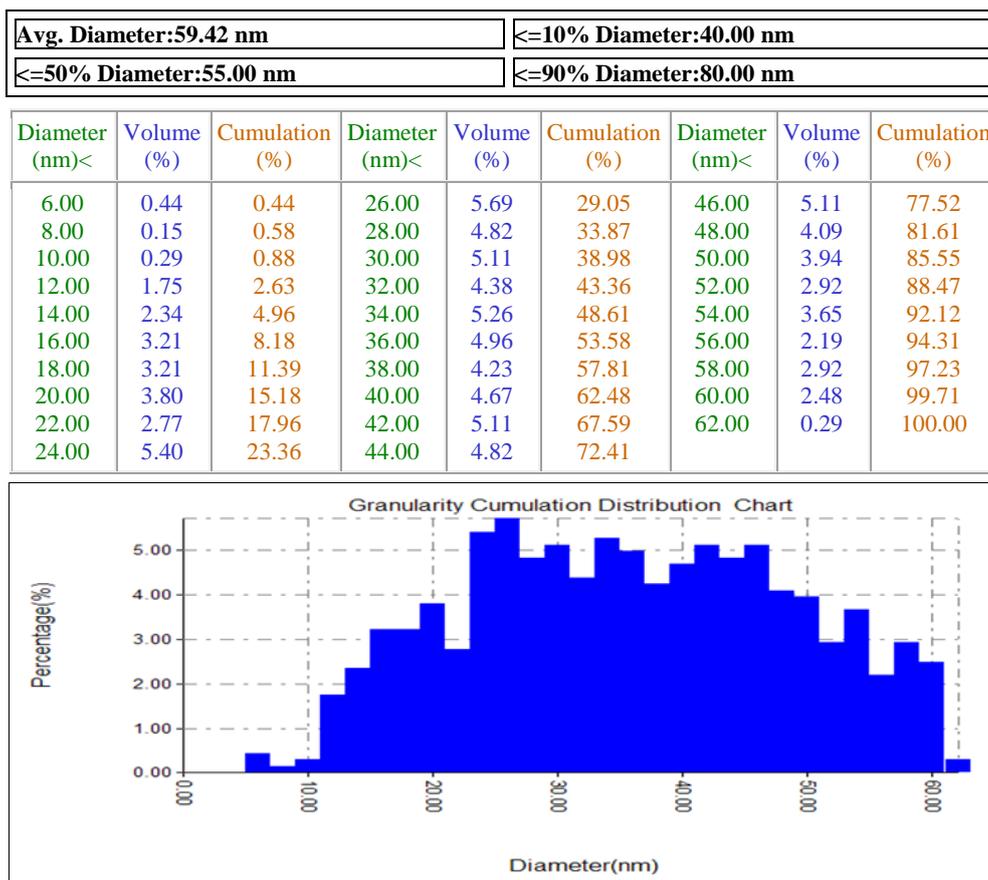


Fig (5) Distribution of statistical size nano particles made up on surface of the sample (b) in fig .(3)

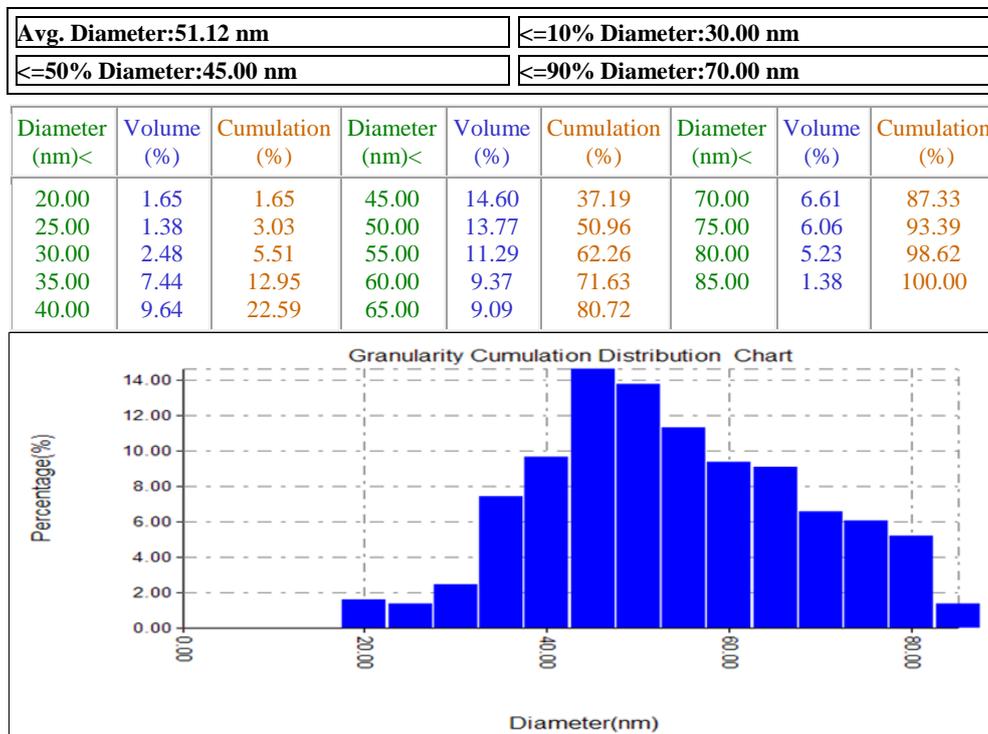


Fig (6) Distribution of statistical size nano particles made up on surface of the sample (c) in fig .(3)

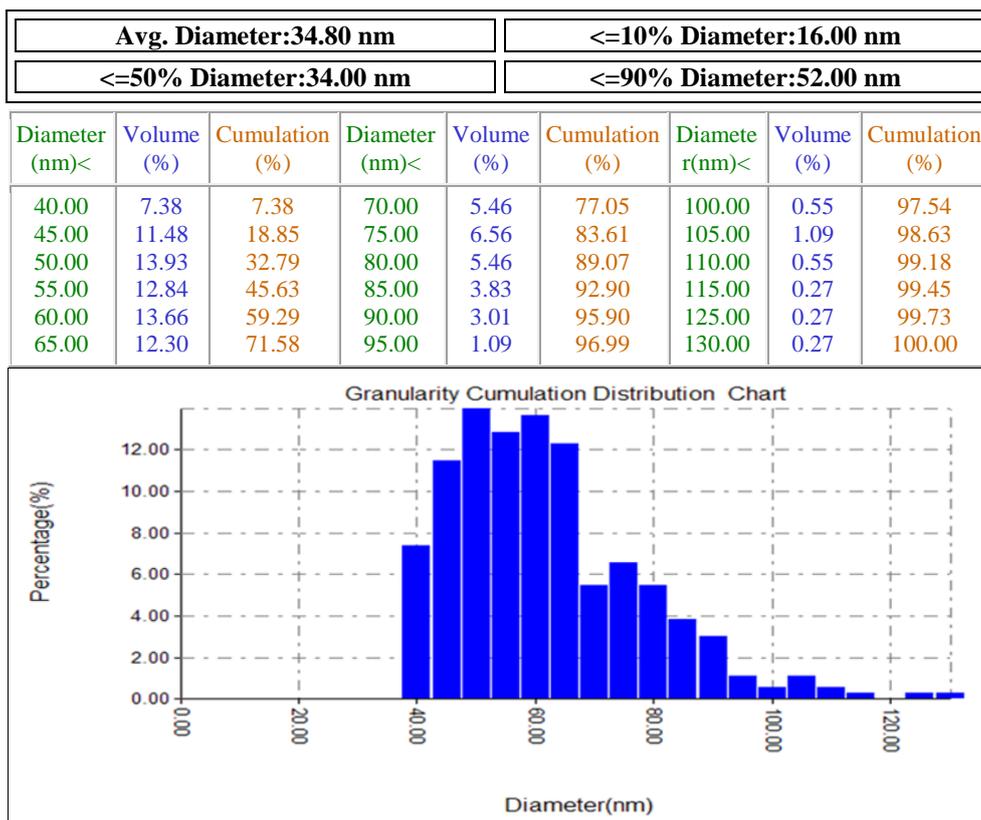


Fig (7)Distribution of statistical size nano particles made up on surface of the sample (d) in fig .(3)

b-The surface morphology by scanning electron microscope (SEM):  
The study of the surface samples is removed by the photochemical etching method showed that the areas appear to dissolve in the middle of the samples and the superficial dimensions [1-D] increase with the intensity of the light on them can be explained in this

areas because the luminescent lenses focus more intensity concentrated light in the center of the focus and thus more chemical removals and when the intensity of the light is greater than a certain limit. The surface liquefaction operations [polishing] start from the lightest area in the middle of the sample as shown in fig (8).

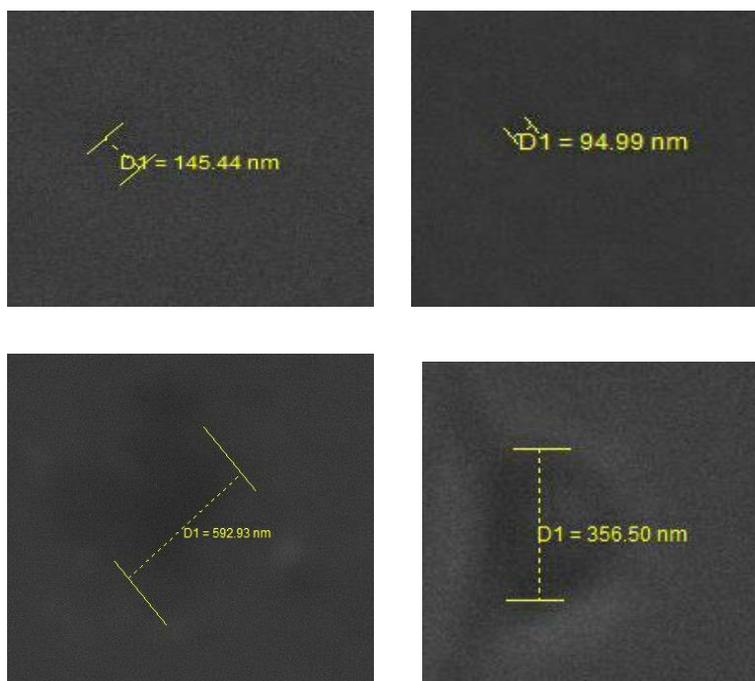


Fig (8) SEM Image for surface morphologies (100) n-type Si samples immersed in (40%)HF acid for constant etching time (60min) prepared by sun light photochemical etching with different power densities: (a)1758 ,( b) 3956 , (c) 6182 , (d) 8902mw /cm<sup>2</sup>

### 2-3. Porosity of (PS)

The porosity ( $P$ ) is defined as the fraction of void space within the porous layer and can be determined easily by weight measurements. The porosity of the layer can be determined by weighting the silicon substrate before and after etching process ( $m_1$  and  $m_2$ )

respectively and again after removing the porous silicon layer by using a molar NaOH or KOH ( $m_3$ ). The porosity is given by the following equation:

$$P = \frac{m_1 - m_2}{m_1 - m_3} \times 100\%$$

The value of porosity of PS sample shown in table(1).

**Table (1) calculated porosity of PS samples prepared with different illumination light.**

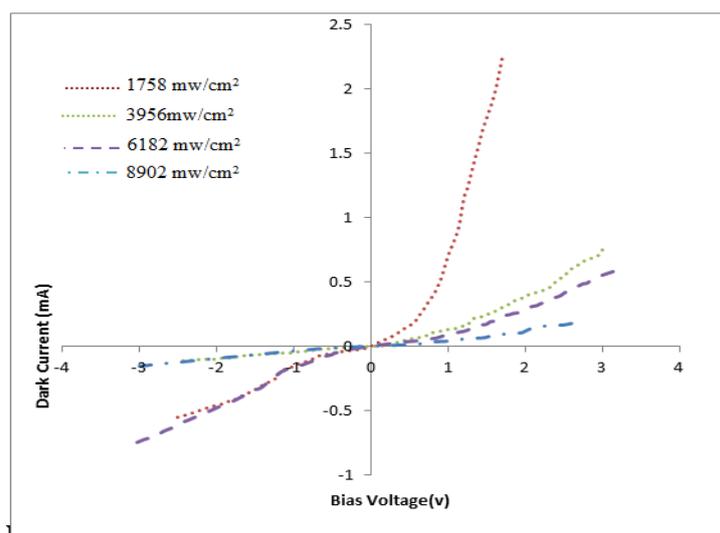
Type wafer	Orientation	resistivity	Time etching	concentration (HF)	Focal Length (cm)	light intensity ( $mw/cm^2$ )	Porosity Of (PS) %
n-type	100	10 $\Omega$ .cm	60min	40%	13	1758	15
n-type	100	10 $\Omega$ .cm	60min	40%	18	3956	35
n-type	100	10 $\Omega$ .cm	60min	40%	41	6182	50
n-type	100	10 $\Omega$ .cm	60min	40%	45	8902	60

The porosity increases with increasing the light intensity as show in table (1), and this lead to decrease the charge carriers (electrons) in the porous Layer. The porosity is strongly depends on the preparation conditions, these conditions include HF concentration, etching time, illumination wavelength and power density of the illumination light[9].

**Electrical Properties:** I-V measurement were recorded for the samples  $AL_{thin}/n-Si/PS/AL_{thin}$

structures fabricated by sun light photo chemical etching (SLPCE) on n- type (100) Si with different light intensities (power densities) (1.7584,3.9564, 6.181875 and 8.9019)  $w/cm^2$  shown in Fig(9) the rectification improves with increasing of light intensity.

This behavior is expected when we probe the change in size of nano structures and porosity .



**Fig (9) The current – voltage characteristics of PS (100) n-type si samples immersed in (40%) HF acid for constant etching time (60min) prepared by sun light photo chemical etching with different power densities:**

(a.1758 , b. 3956 , c. 6182 , d. 8902)mw /  $cm^2$

### References

- [1] A .f. Mohammad," study of laser Effects on porous silicon properties " Master Thesis/ department of Applied sciences/ University of Technology(2006).
- [2] S. Jan. S " production of porous silicon by laser – Induced Etching "Master Thesis/ department of Applied sciences / University of Technology(2006).
- [3] Mukarram Abdul- mutalib Al-gaafar, "production of porous silicon studying Its Electrical properties". Master thesis /Department of laser and optoelectronic Engineering , University of Technology (2007).
- [4] V. Lehmann, and U. Gosele"porous silicon formation a quantum wire" Appl. Phys. Lett. 58 (1991) 856.
- [5] A.M. Alwan, "Electrical properties of porous silicon prepared By photochemical Etching". XXII Regional conference on solid state science and technology, 18-21, (2005).
- [6] H .Asker .K," preparation and study of porous silicon layers from n-type silicon wafers using different etching techniques "PhD thesis college of science , University of Baghdad(2010).
- [7] Jeyakumaran N, Natarajan B, Ramamurthys. And vasu V, " structural and optical properties of n-type porous silicon – effect of etching time", I J NN, Vol .3,pp. 1, , 2007
- [8] Y.Z. Dawood " preparation of porous silicon and It's Application in solar Cell "Master Thesis

Department of Applied Sciences/ University of Technology (2009).  
[9] R.M. Shehab " preparation and Characterization of Nano Porous silicon for chemical Detection" Master thesis Department of Applied Sciences,

University of Technology(2015). [10] B.G. Rasheed, "production of porous silicon by laser - induced etching", India,(2001).

## تحضير شرائح السليكون المسامي النانوي باستخدام ضوء الشمس

اسماعيل خليل جاسم<sup>1</sup> ، احمد ياس خضير<sup>1</sup> ، حسن عسكر كاظم<sup>2</sup>

<sup>1</sup>قسم الفيزياء ، كلية التربية للعلوم الصرفة ، جامعة تكريت ، تكريت ، العراق

<sup>2</sup>مديرية تربية كركوك ، وزارة التربية ، كركوك ، العراق

### الملخص

تم تحضير طبقات من السليكون المسامي بطريقة التتميش الكيميائي الضوئي. استخدمنا ضوء الشمس وللمرة الاولى كمصدر للطاقة الضوئية بدلاً عن مصادر الطاقة الصناعية كالليزر والهالوجين ومصابيح التلستون التي كانت تستخدم لتحضير طبقات السليكون المسامي. السليكون المستخدم من نوع n-type (100) وبمقاومة (10Ω.cm) وحامض بتركيز (40%HF). تم الحصول على شدة اضاءة مختلفة  $2\text{mw/cm}^2$  (1758,3956,6182,8902) باستخدام عدسات لامة بأقطار مختلفة (40,60,75,90)mm وذات ابعاد بؤرية (13,18,41,45)cm على التوالي ،وزمن تتميش ثابت (60min) . تم استخدام مجهر القوة الذرية (AFM) والمجهر الضوئي الماسح (SEM) لدراسة طبيعة سطح طبقات السليكون المسامي وبينت نتائج السطح تشكيل طبقات سليكون مسامي كمجاميع من المسامات والبلورات النانوية وبمختلف الاقطار وبأشكال تموجيه متوزعة على السطح المسامي وذلك بازيادة شدة الإضاءة. سمك الطبقة المسامية بمدى من (3.39,3.06,3.79and50.06)nm وبمعدل قطر للجسيمات (84.37,59.42,51.12,34.8)nm على التوالي. خصائص تيار – فولطية على النبايط (AL<sub>thin</sub>/n-Si/PS/AL<sub>thin</sub>) المصنعة من السليكون المسامي المحضر بطريقة التتميش الكيميائي الضوئي باستخدام ضوء الشمس اظهرت خصائص تقويمية تزداد مع زيادة شدة الاضاءة.