### **Light-Induced Etching of Silicon**

Dr. A.M. Ahmed\*

Alwan. M. Alwan\*

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Abstract

In this work, an ordinary light is used for photo-chemical etching of n-type silicon wafer in HF solution. Scanning electron microscopy is used to monitor changes in surface morphology produced during the etching process. Uniform porous layer has been observed for various irradiation time. Our technique offers a great controlling parameter on the porous layer uniformity compared with the porous layer achieved by using a laser beam. Electrical properties and porous layer thickness of the photo produced layer have been studied.

Keywords: photo-chemical etching, porous silicon.

القشط المحتث بالضوء للسليكون

الخلاصة في هذا العمل تم إستخدام الضوء الاعتيادي في عملية القشط الضوء - الكيمياوي للسليكون في حامض الهيدروفلوريك تسم إستخدام المجهر الالكترونسي الماست لمراقبة التغيرات الطوبوغرافية الناتجة على سطح السليكون تم الحصول على طبقة سليكون مسامي منتظم باستخدام الضوء الاعتيادي بالمقارنة مع الليزر المستخدم في بحوث أخرى

#### **1-Introduction**

The formation of porous silicon in HF acid with out external biasing was first reported by Noguchi and Suenume [1] 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 [2]. The detachment of one silicon atom can be expressed as follows

 $Si_{H_2} + 4F^+ + 2H^+ + h^+ \rightarrow Si_{H_2} + Si_{H_2} + H_2 \uparrow$ 

where holes help to substitute fluoride for hydrogen on the passivated silicon surface, the initial stages of etching on the flat surface can be understood in terms of band bending at the silicon/electrolyte interface as discussed for example, by Beale et al [3] and Gerischer and Co-workers [4,5]. In n-type silicon band bending in the space charge region forces holes toward the surface where, according to the above reaction, they initiate etching of silicon and porous silicon formation. If electron-hole pairs are created by photon absorption deep in the bulk (below the space charge region), the carriers will thermally diffuse through the crystal, some will reach the space charge region at the front side charge separation will then occur and again reaction will ensue.

In laser-induced etching a power densities in the range (0.55 to  $5.5 \text{ w/cm}^2$ ) from He-Ne laser are suitable to produce porous thin films within irradiation times (2-20 min) [6], while in [7,8] the etching power densities varied from (8-20 w/cm<sup>2</sup>)

\*Applied Physics Department, University of Technology Baghdad.

Corresponding author. Tel: 009641 7193599, E-mail address: phys. Tech-04@yahoo.Com.

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2412-0758/University of Technology-Iraq, Baghdad, Iraq This is an open access article under the CC BY 4.0 license http://creativecommons.org/licenses/by/4.0 from Nd:YaG laser which provide a resonance condition in etching, a thick porous layer (120  $\mu$ m) was obtained at an irradiation time about 30 min.

The present work reports preparation of porous silicon by light-induced etching process by using a high power density photo source (100w) quartz Tungsten halogen lamp integral with the dichroic ellipsoidal mirror instead of laser source and study the surface morphology of the etched surface. The I(V) characteristics and reaction rate, are studied.

### **<u>2. Experimental Procedure</u>**

Fig.1 shows a schematic diagram of experimental set-up for light-induced etching process. A commercially available mirror-like ntype (111) oriented wafer of resistivity

$$(r_1 = 3.25 * 10^{-4} \Omega.cm)$$
 was

rinsed with cetonic and а ethanol to remove impurities and with dilute (HF) to remove the native oxide and then immersed in electronic grade (40%) 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). In this electrode less photochemical etching process, there was no applied bias. The light beam of quartz Tungsten halogen integral with lamp dichroic ellipsoidal mirror has been focused on a silicon wafer to a circular spot  $(1.13 \text{ cm}^2)$  area, thereby light power density of order of  $(88.5 \text{ w/cm}^2)$ , the distance between the halogen lamp

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and the wafer about (4 cm). The spectral range of the halogen lamp is given in Fig.(2) Bubbles were observed during the etching process. Wafers were etched for irradiation times ranging from (5-120 min), 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.



## Fig.1. Experimental set-up for Light-induced etching.

The surface morphology of the etched surface was performed by using scanning electron microscopy (JSM-5510). For electrical measurements an aluminum electrodes were deposited on the front and back surface of light etched area. The I(v) characteristics were performed in dark rooms and under day light illumination at room temperature by using two Keithly

(616) digital electrometer and (50V) power supply



Fig.2 Spectral distribution of quartz Tungsten halogen lamp.

# 3- Experimental results and discussion

#### **3-1 Surface morphology**

Fig.(3) shows SEM micrographs of an ordinary light etched n-type silicon wafer produced at a constant light power density of about (88.5 w/cm<sup>2</sup>) for different irradiation time. Fig.(3.a) represent an atomically flat silicon surface just before the etching process. For an irradiation time (10 minutes), a disconnected pits are observed and these pits formation commenced with the initiation of etching process on silicon wafer, an average pits diameter of about (100 nm) with wall between pits less than (300 nm), as shown in Fig.(3.b). As the etching process proceeds for (90 minutes), a uniform porous structure with pores diameter mostly in the range (350 nm) while the walls between two pores of nearly less than (80 nm) as shown in Fig.(3.c).

The ordinary light in this work for the etching process has a uniform intensity distribution compared with the Gaussian distribution of the laser beam used by others [1, 6, 7, 8] for the etching process. Therefore, a uniform porous layer thickness of specific crystallite size distribution can be expected here.

Our observation for the produced porous structure reveal existence of small difference in the porous thickness and that is due to the effect of the spectral distribution of the ordinary light consisting many wavelengths. These wavelengths have a various penetration depths [21] in the silicon which leads to produce a porous layer with a small difference in its depth.



10kU ×50,000 0.5mm 0000 10 30 SEI (b) 1844 X18,000 Two 0000 29 30 SEL

(c) Fig.3. SEM images of the silicon surface etched by (88.5 w/cm<sup>2</sup>) power density from halogen lamp, for irradiation times of (a) just before the irradiation, (b) 10 min, (c) 90 min and wafer resistivity r=3.25 x 10<sup>4</sup> W.cm.

## 3-2 Current-Voltage Characteristics

Fig.(4) shows the I(v) characteristics as function of the etching time ranging from (5-120 min) under a fixed irradiation power density of (88.5 w/cm<sup>2</sup>). A Schottky-like junction is formed on the surface of the light etched layer, the rectification ratio, ideality factor and barrier height is varied with the etching time as shown in table (1).

Table (1). Results obtained from I(v)

Etchin	Rectificati	Ideality	Barrier
g time	on ratio	factor	height (f
(t)	(a)	<b>(n)</b>	bn) (eV)
(min)			
5	25	9.2	0.685
10	11	8	0.686
20	6	15.3	0.67
30	16	9.5	0.66
40	34	9.6	0.656
60	240	8.77	0.65
90	16	10.2	0.686
120	62	6	0.678





**(b)** 

Fig.4 Current density-voltage characteristics of light etched surface at a various irradiation times of (a) 5 min, 20 min, 30 min, 40 min, 90 min, (b) 60 min, 120 min and wafer resistivity  $(r_1 = 3.25 * 10^{-4} \Omega.cm)$  rectification ratio at (5 v)

The resistivety of the etched layer in the thickness direction is obtained from the slop of the forward I(v) characteristics in the high current regime which gives a straight line, Fig.(5) shows this resistivity as a function for etching time. It is clear that the resistivity of etched layer has the high resistivity of  $(10^4 - 10^5 \Omega.cm)$  in comparison with the resistivity of the bulk substrate of  $(10^{-4} \Omega.cm)$ . Eng.& Technology, Vol.25, Suppl.of No.3, 2007

The values of ideality factor and Barrier height is comparable to that obtained by D.B.Dimitrov[9] in electrochemical etching. the rectification ratio at (5 v) is also in the same range as that obtained by M.B. Chorin [10]. While the resistivity of the porous layer that occurs at the etching time (30, 40, 90 min) has the same order of magnitude  $(10^4)$  $\Omega$ .cm) to these obtained by T.Unagami [11] in electrochemical etching process.



Fig.5 Resistivity of light-etched layer (L.E.L) as a function of irradiation time, wafer resistivity  $(r_1 = 3.25 * 10^{-4} \Omega.cm)$ .

Fig.(6) shows the photo current of the light etched wafer as a function of etching times at a various reverse biasing voltage. The photo current  $I_{ph} = I_b - I_d$  where  $I_d$  is the dark current and I<sub>b</sub> is the current obtained with the room light on, this photo response at etching times (30, 40, 90 min) is due to the hetrojunction that formed between the porous layer and the silicon substrate where the porous silicon energy gap is greater than n-type silicon due to the quantum confinement effects [12,13].



Fig.6 Photo current at daylight illumination as a function of irradiation time, wafer resistivity  $(r_1 = 3.25 * 10^{-4} \Omega.cm)$ .

#### 3-3 Etching rate

the thickness of the lightlayer etched is obtained by subtracting the maximum depth of upper interface from the the maximum depth of lower interface. Fig.(7) shows the etching layer thickness with the irradiation times from (5 to 120 min) for wafer resistivity  $\rho = 3.25^{-4} \times 10^{-4} \Omega$ .cm. The rate displayed in Fig.(8) is derived from data in Fig.(7) by dividing the layer thickness by the etching times as in [6] to obtain the reaction rates expressed in nm/sec. The low resistivity, high doping rate wafers are etched easier where according to a simple depletion approximation, the thickness X<sub>o</sub> of the barrier can be estimated from the following equation.

$$X_{o} = \sqrt{\frac{U \in O}{2pN_{o}q}}$$

where U is the height of the barrier (schottky barrier between wafer and solution) and  $N_D$  is the doping concentration. For our samples with U=0.5eV and  $N_D \sim 10^{19} \text{cm}^{-3}$  the depletion layer thickness are (0.6-1 nm) so that the holes are lass likely to enter the pore wall, so that is does not fill the core of the wall and holes penetrate into porous structure [14].

Fig.(7) exhibits a relation between the irradiation time and the porous layer thickness. We can easily distinguish two regions: one at short irradiation time in which the thickness dependence linearly on the time. This behavior is similar to the result of Y. Arita et al [15], who obtained (1-12 µm) porous layer from an electrochemical etching of low resistivity n-type silicon wafer and low anodization time (15-70 sec).

At long irradiation time, the thickness of the porous layer increased with the square root of the time (region two). This results in this region is in a good agreement with that obtained by Y.Watanabe et al [16], who employed an electrochemical etching for a low resistivity n-type silicon wafer and anodization time (1-20min) to obtain layer. [120 um] porous This nonlinear relation between the irradiation time and porous layer thickness is due to reduction of the photo generated carriers concentration in the porous region and then increased this of concentration in the bulk. The etching rate tends to saturate at a certain level in spit the increased in the irradiation time as shown in Fig.(8).



etching time /sec.

Fig.7 Etching layer thickness as a function of irradiation time, wafer resistivity  $(r_1 = 3.25 * 10^{-4} \Omega.cm)$ .





Fig.8 Rate of light-induced etching process as a function irradiation times, wafer resistivity  $(r_1 = 3.25 * 10^{-4} \Omega.cm)$ .

Beale et al [3], Smith and Collins [2] and Lehman and Föl [17,18] have discussed how electric field in electrochemical etching effects focusing can lead to preferential transport of holes to the bottom of pores rather than the pore walls. This leads to preferential etching at the bottom of pores. In the present work, there is no applied potential, therefore, the only source of electric field inside the silicon would be from band bending. While there is certainly a band offset between bulk silicon and porous silicon layer [19], Romstand and Veje [20] have suggested that no band bending occurs at the porous silicon/bulk silicon hetrojunction, i.e there is an a abrupt transition. Regardless of wither there is band bending or an abrupt transition at the interface, the band offset, which results from quantum confinement, is sufficient to lead to a focusing effect. This is described by Lehman and Gösele [19] who have discussed quantum models that invoke the change in the electronic structure of porous silicon relative to bulk crystalline silicon to explain the preferential etching that occurs at the bottom of the pores. This quantum confinement effect is shown in the etching times (30, 40, 90 min), which results porous silicon with smallest crystallites in etching cycle.

The size of the silicon structures found in micro porous silicon is irregular, this is due to the photon absorption of the ordinary etching light with it, larger band width from near infrared region to visible as shown in the Fig.(2), where the peak of the lamp emission is around (  $\lambda = 0.9 \mu m$ ) and this will provide a good absorption due band to band transition and also due the absorption the impurities by especially at the larger wavelengths. The absorption length which depends on the wave length of the light is varied from (sub um to few um) [21], 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.

### **4-** Conclusion

An ordinary light was used for photo-chemical etching process and this technique offers the production of porous silicon of uniform thickness. The electrical properties etched layer show a rectifying behaviour similar to that of schottky like junctions with porous layer resistivity eight order of magnitude higher than the silicon wafer. The AL/Ps/n-si/AL structures shows a photo response similar to a simple photodiode.

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