

The Effect Of Thermal Oxidation Time On The Structure And Influence On Optical Properties For Porous Silicon Prepared By Photo Electrochemical Etching.

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Abstract

The morphological properties of the freshly and oxidized porous silicon at oxidation time (60, 90) sec were studied. A blue emission from PSi can be seen with eyes after thermal oxidation because the increasing of energy gap due to decreased silicon column (nano particles). Pore size and shape of n-type wafers are estimated and correlated with optical properties before and after rapid thermal oxidation (RTO).

Keywords: porous silicon, oxidation, optical properties.

تأثير زمن الأكسدة الحرارية على الخصائص التركيبية وتأثيرها على الخصائص البصرية للسيليكون المسامي المحضر بطريقة الكهروكيميائية

الخلاصة

في هذا البحث تم دراسة تأثير الأكسدة الحرارية السريعة على السيليكون المسامي المحضر بطريقة القشط الكهروضوئي كيميائي على طوبوغرافية السيليكون المسامي . وقد تم من خلال صور الماسح الإلكتروني حساب قطر وتوزيع المسامات وحساب حجم العمود الفاصل بين المسامات قبل وبعد عملية الأكسدة الحرارية كما تم قياس الخصائص البصرية والكهربائية قبل وبعد المعالجة الحرارية

Introduction:

Porous silicon (PSi) is an interesting material for making integrated optical devices because it is easy modifiable optical properties. The photo oxidation process has been already studied by series of researches [1]. Transformation of porous silicon into oxidized porous silicon (porous silica) modifies of course the physical characteristics and optical properties of porous silicon layer [2-3], and also allow light emission in the visible wavelength range. However, the light emission of porous silicon generally appearing the red-

green light range, but not in the blue light emission, it is difficult to achieve [4].

Furthermore, the surface of as-etched porous silicon can be hydrogen, so-called H-passivated, so that the PL intensity of porous silicon decays rapidly if it is exposed to atmospheric air for a period of time, which will lead to continuous changes of its electrical and surface properties with time [5]. In order to stabilize the physical and optical characteristics of as prepared porous silicon several techniques have been developed. Among them, the

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oxygen passivation by the formation of SiO₂ layer on Phi is one of the most effective router. Various oxidation approaches such as chemical oxidation [6], anodic furnace oxidation [7], conventional oxidation [6], rapid thermal oxidation [8], ageing oxidation [9] and PECVD method [10], were used to generate a more stable O-passivated surface.

Moreover, these Oxidation methods can also improve the efficiency of light emission and blue shift [11]. In these papers, we reported the effect of (RTO) on the morphological and transport mechanisms of porous silicon for fresh and partially oxidized n-type porous silicon. It was found that the ROT will modify the morphological and electrical properties.

Experimental

Crystalline wafer of n-type Silicon with resistivity of (1.5-4.5) Ω.cm, 508 μm thickness, and (111) orientation were used as starting substrates. The substrates were cut into rectangles with areas of (1×1.5) cm². The native oxide is cleaned in a mixture of HF and Ethanol (1:4) to obtain 10% HF concentration. Photo-electrochemical etching then performed in HF of 47% concentration and Ethanol (1:1) at room temperature the electrical circuit is complete after putting a Pt electrode in parallel way to achieve the homogenous PSi layers. Current density of about (20 mA/cm²) applied for (15min), light source is consisted of one commercially available CW diode laser with power (2W) and wavelength (810nm), (fig.1) shows the schematic diagram of PEC system. The RTO system is consisted of the following: (1) a tungsten halogen photo optic lamp type (OSRAM 64575) with power 1000W

based on ceramic base. We put a parabolic reflector like half circuit under the lamp to increase the heating efficiency and (2) a quartz tube with 2 cm

diameter opening from two sides to allow air to input (dry oxygen source) the quartz tube was put attached with halogen lamp to obtain the temperature 750 °C, (fig.2) shows the photographic image for oxidation system. The samples were characterized by scanning electron microscopy (SEM) measurements were carried out in the School of Physics / Nanostructures and Optoelectronics Research Center (NOR)-lab, University Sian Malaysia (USM).

Result and Discussion

Fig.3 shows the scanning electron microscopy (SEM) of the as prepared and oxidized porous silicon etched from n-type crystal silicon, of sample (fig. 3,a,b,c) where fresh sample (A) represented in (fig.3,a) sample (B) represent sample after oxidation time 60 sec in (Fig.3b) and (Fig.3c) represent sample (C) where oxidation time 90 sec. These micrographs allowed the size and density of porous to be estimated. In fact; these values contained an estimate error because of the micrograph contrast. The pore size was measured directly from these micrographs and its distribution was deduced in (fig.4) it could be seen that the porous layer was composed of a pore network (in black) separated by silicon crystallites (in white). For sample A, pore size was varied from (1.6-5.3) μm center around a mean value equal to (3.3), this distribution was almost symmetrical due to Gaussian distribution for the laser beam where the laser intensity at the center larger than at the edge [12].

Fig.4 shows that the distribution with allow root mean square the revealed an homogeneity in pore size values this distribution was almost symmetrical for sample B and C but different in mean

value, the value corresponding to maximum after oxidation pore size decreased and became (3 μ m) for sample (B) and (2.6 μ m) for sample (C), from SEM image in (fig.3) we could see that after oxidation the value of roughness are always lower, this decrease in roughness after oxidation was also observed by *pap et al.* [13] and *chrier et al.* [14]. The wall size, which separate porous, also can be evaluate, that mean the energy gap increased after oxidation according to following relation [15]:

$$Eg^* = Eg + 88.34/L^{1.37} \dots\dots(1)$$

Where Eg^* : is the energy gap for PSi layer in (eV), Eg : is the energy gap for silicon substrate the energy gap of in (eV) and L (nm) is the nano crystallite size. Porous did not collapse because of the initial porosity which define as a fraction of voids inside the PSi layers [16], the density was nearly the same before and after oxidation and it was about 16×10^6 pore/cm², the pore density was nearly the same and pore size decreased, so the porosity after oxidation of course lower than the porosity before oxidation, because the decreasing in a fraction of voids after oxidation due to oxide growth inter PSi layer. Moreover table (1) shows pore morphology estimated from (SEM) micrograph of porous silicon before and after thermal oxidation. The difference in band gap after rapid thermal oxidation caused to change the optical

and electrical properties [17]. Fig.5 shows the electrical behavior for as prepared and after thermal oxidation. The J-V curve of fresh heterojunction shows the rectification behavior due to the formation of an isotope Heterojunction with low rectification factor (About 5 at 5 V), since

PSi is reported to be n-type when it is fabricated from n-type substrates [18].

The J-V characteristics of Al/porous silica/n-silicon/Al sandwich structure which contain PSi such as shottky diode generally is determine depending on the forward current – voltage characteristics, Table (2) shows the J-V characteristics after oxidation. We can obvious that the dark current is increased with oxidation time, it could be from a defects formed by no sympathy silicon – oxygen structure in a very thin oxide and they would act as tunneling centers [19]. The rectification factor is increased to 13.5 at 60 sec of oxidation, this increase in rectification factor is attributed to the formation of a thin oxide layer between Al metal and Si. This interfacial layer introduces a MIS structure, which in turn leads to a decrease in reverse saturation current and hence increases the rectification factor. After the time 60 sec rectification ratio will be decrease, it could be from the defects.

Fig.6 shows the Photo current at reverse bias voltage (0.2-5)V, we observed increase photo current with increasing oxidation time when it shorter than 60 sec that because after 60 sec of RTO treatment, surface states will replace unstable hydrogen-passivated surface; therefore, the photo carriers generated in Si wires are greatly increased and so increase the

photocurrent. As the oxidation time 90 s photo carrier will be decreased that result in thinner wire size and therefore longer energy band gap of PSi, the absorption energy of the PSi layer is further away from the main portion of tungsten lamp is in the range of 400-3000 nm with the main portion at around(875 nm) [18]. The contribution of Al/PSi contact is not significant for fresh junctions, because the barrier height which is calculated from the semi-log J-V curve (not shown here) is (0.7742 eV), with

neglect of series resistance. The barrier height is raised to (0.7769 eV) at (60 sec) of oxidation time. This increasing in barrier height attributed to the formation of MIS structure at Al/PSi contact by producing an oxide interfacial layer after thermal oxidation. The increasing in the barrier corresponds to an increase in rectification characteristics with bit-by-bit Lessing in the current values due to the increase in series resistance of the junction [19]. The existence of the oxide layer and the increase of the PSi resistivity (due to band widening) will result in an increase in series resistance, i.e., which is indicated by an increase in the slope of the forward current. *Balagurov et al.* [20] exhibited similar results for their Al/PSi/c-Si heterojunction after oxidation, in which they attributed the improvement in the J-V characteristics of the oxide samples to the substrate potential barrier.

Conclusions

We studied the physical characteristics of porous silicon layers before and after oxidation. The pore size and column size decreased after oxidation. However, the pore density conserved, the porosity decreased after oxidation, photo

current will be increased about 7 order after oxidation. We can suppose that the oxidation of PSi layers is a good way to obtain lower optical loss of PSi photo detector by reducing both volume scattering and absorption in the near infrared wavelength range.

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References

- [1] S. shih, C. Tsai, k. H. jung, j. c. campbell, D. L. Kwong, "porous silicon Photoluminescence through oxidation", *App. phys*, 60 (5) (1992) 633.
- [2] L. T. canham, "properties of porous Silicon," *INSPEC*, (1997) 158-161.
- [3] W. Theiss, G. Amato, C. Delerue, H. J. Von Bardeleben (Eds.), "structural and Optical properties of porous silicon Nanostructures", Gordon & Breach, 1997.
- [4] W. Liu, M. Zhang, C. Lin, Z. Zeng, L. Wang, P. K. Chu, *Appl. Phys. Lett.* 78 (1) (2001) 37.
- [5] T. V. Torchynska, M. K. Sheinkman, N. E. Korsunskaya, L. Y. Khomenkovan, B. M. Bulakh, B. R. Dzhumaev, A. Many, Y. Goldstein, E. Savir, *Physica B* 273-274 (1999) 955.
- [6] N. Rigakis, J. Hilliard, L. A. Hassan, J. Hetrick, D. Andsager, M. H. Nayfeh, *J. Appl. Phys.* 81(1) (1997) 440.
- [7] R. Boukherroub, D. D. M. Wayner, D. J. Lockwood, *Appl. Phys.* 81 (4) (2002) 601.
- [8] S. S. Chang, A. Sakai, R. E. Hummel, *Mater. Sci. Eng.* B64 (1999) 118.
- [9] Y. Fukuda, K. Furuya, N. Ishikawa, T. Saito, *J. Appl. Phys.* 82 (11) (1997) 5718.
- [10] Y. Zhao, D. yang, D. Li, M. Jiang, *material*

science,116(2005)95.
 [11] R. Kumar, Y. Kitoh, K. Hara, Appl. Phys, 63 (22) (1993) 3032.
 [12] K.cheah and c.choy Appli.phys, 61(1995)45.
 [13]A.E.Pap,K.KordasToth Fg.Appl.phys ,86(2005)041501.
 [14]J.Charrier, V.Alaiwan , P.Pirasteh ,A.Najar,M.Gadonna.Appl .sience, 253(2007)8635.
 [15]P.A.Kohi,J.Res.Develop,24 (1998) 629.
 [16]L.T.canham,"propertiesof porous silicon",INSPEC (1998)
 [17]W.Hyoug lee,C.hoochon lee and J.Jang,J.Jang .J. Nano-crystalline solids , 198(1996)911.
 [18] A.M.Alwan, O. A .Abdulrazaq, J. moderen physics .lett.22, 417(2008)
 [19]M.K.lee,Y.H.wang,C.H.Chu, Quantum Electronics, 33(12) (1997)199.
 [20]L. Balagurov, S. Bayliss, S. Andrushin,A. Orlov, B. Unal, D. Yarkin and E.Petrova,Solid-State Electronics 45, 1607 (2001).

Table (1); Illustrated the structural properties of formed porous silicon layer at constant parameters. Current density (20mA/cm²), etching time (15 min) before and after rapid thermal oxidation.

Oxidation time (sec)	Pore size (µm)	Well size (µm)	Energy gap (eV)	Porosity %	Pore Shape	Roughness
fresh	1.6-5.3	.023-01	2.3	55	Triangular	High
60 sec	1.3-5	0.013-0.46	3.75	27	cylindrical	Low
90sec	1.3-5	0.01-0.2	4.88	16	cylindrical	Smooth

Table (2); Shows the electrical properties after and before oxidation.

Oxidation time (sec)	Rectification ratio	Saturation current density (µA/cm ²)	Ideality factor (n)	Barrier height (eV)
Fresh	5.144969	1	5.09	0.7742
60	13.51351	0.9	2.1	0.7769
90	9.578947	1.1	2.7	0.7718

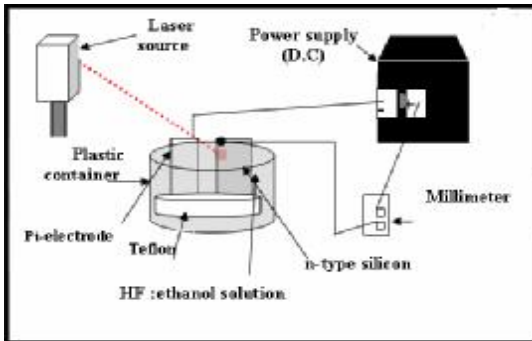
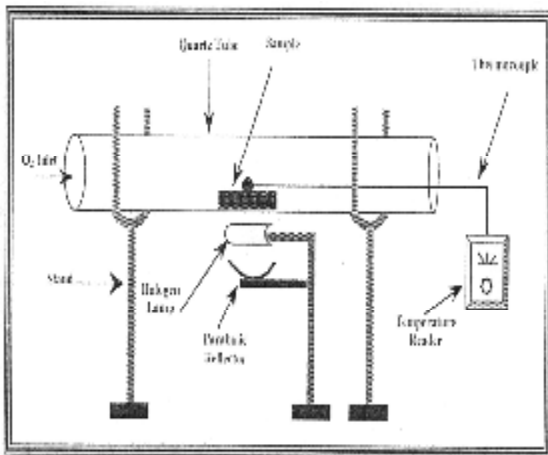


Fig.1; schematic digram depicts the PEC



proces.

Fig. 2; Shows the Schematic diagram for RTO system.

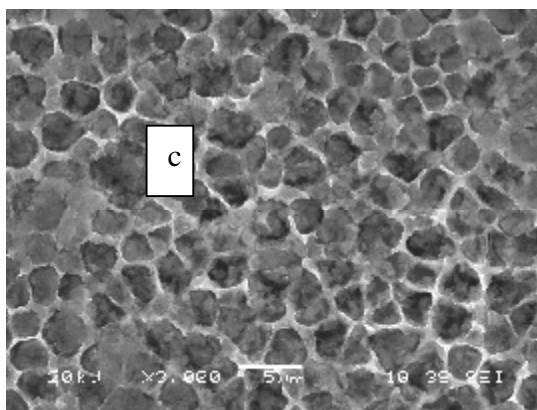
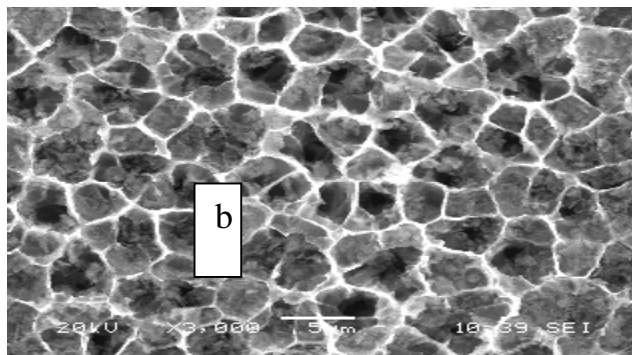
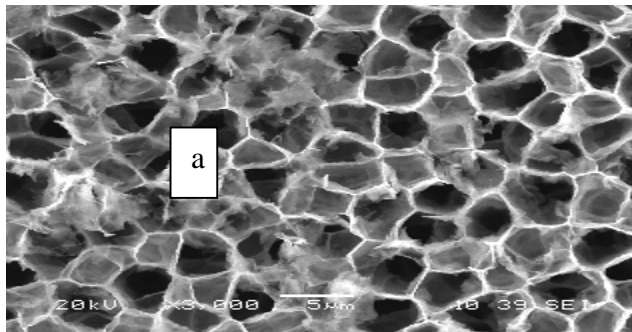


Fig.3 ; Shows SEM micrographs of a surface of(a)as prepared porous silicon(b)60 sec oxidized P*Si* and (c)90 sec oxidized P*Si*

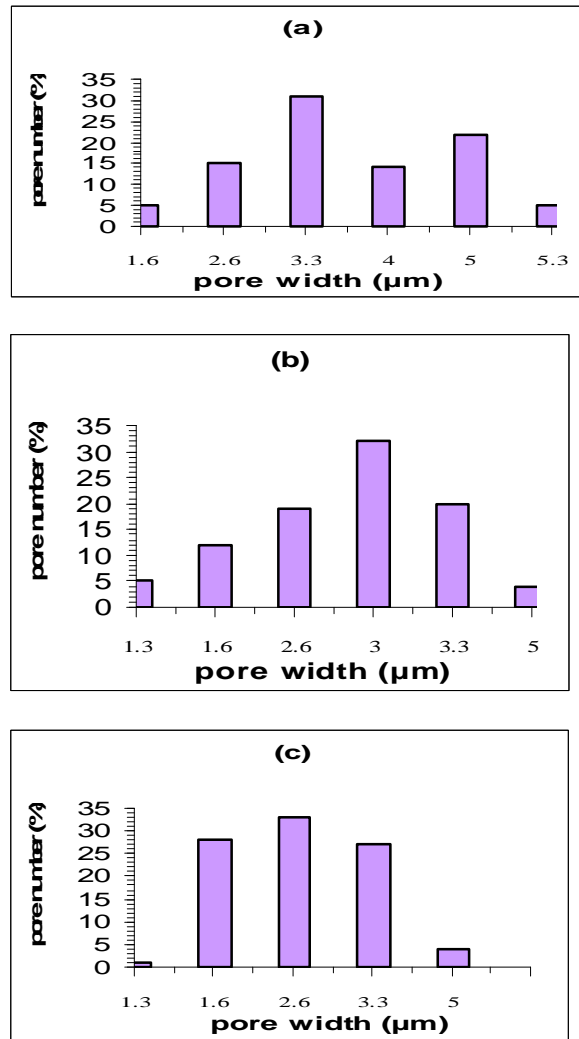


Fig.(4); Distribution of pore size of(a)as prepared porous silicon (b)60 sec oxidized PSi and (c)90 sec.

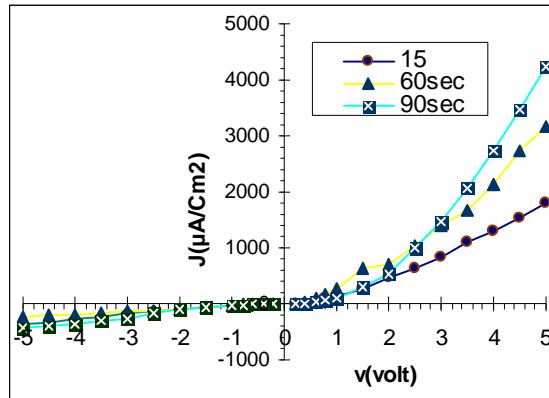


Fig.5; J-V characteristics of Phi/n-Si isotope heterojunction.

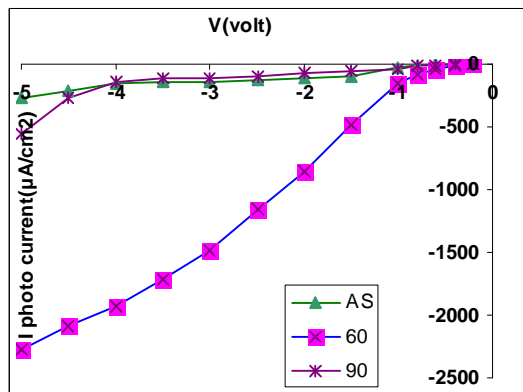


Fig.6; the photo current as a function