Structural and Optical Properties of Multiple Etched Silicon-Porous silicon Structure

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Abstract- **Starting from p type <100> silicon of resistivity 3-10 ohm-cm single etched and multiple etched PS layers were fabricated by using metal induced vapor phase stain etch method. Experimental layers of different porosity and thickness were formed by varying the etching parameters. Optical microscope imaging had been used to visualize the produced structure. The optical characterizations of fabricated structures were done by using reflectance spectra and photoluminescence (PL) spectra. The bonding structure had been investigated using Fourier Transform Infrared (FTIR) spectroscopy. The results were analyzed to find whether the multiply etched layers produce periodic multilayer porous structure. From reflectance results it was found that as the number of etching steps were increased the reflection decreased. This might be due to aperiodic layer formation which had reduced reflection. This result indicated that these structures might be used as antireflection coating.**

Keywords- ***porous silicon, metal induced vapor etch, multilayer, reflectance, photoluminescence, FTIR.***

I. INTRODUCTION

The remarkable characteristics of visible photoluminescence of porous silicon have made it a very interesting and promising material [1]. Due to its particular structure and surface reactivity, porous silicon has stimulated much research and many applications in different fields. One of the main obstacles in the application is that the luminescence intensity from porous silicon had not been strong enough and it has broad band luminescence (FWHM typically 150nm) [2]. Since control of size distribution within a layer of porous silicon is extremely difficult, an alternative method is needed to reduce the luminescent bandwidth. The porous silicon multilayer structure has been fabricated to solve this problem [2,3]. The reflectivity of these multilayers can also be controlled by choosing proper thickness and refractive index of each layer [2-5]. Porous silicon micro cavity structure has multiple luminescence peaks with FWHM as narrow as 3 nm [2]. These narrow peaks are highly sensitive to any slight change in refractive index.

Conventionally porous silicon multilayer structures are fabricated using electrochemical etching of silicon. Periodic current density square pulse is applied during the electrochemical dissolution process [2-6]. A regeneration time at zero current is incorporated into the formation scheme to allow the system to reach equilibrium before the formation of another porous silicon layer. The porosity and thickness of each layer is controlled by the current density and time. No other fabrication methods are used to design multilayer.

Vapor phase etching is a very simple and low cost procedure to fabricate porous silicon [7-9]. But no work till now has been reported on the effect of multiple etching of silicon using this method.

In this work an attempt was made to fabricate porous structure by single etching and multiple etching of p type silicon wafer using metal induced vapor phase stain etching. The structure visualization was done by optical microscope. The optical properties of fabricated structures are investigated using reflectance and pl measurement. The results were analyzed to determine whether multiple etching produces periodic multilayer structure. The bonding

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structure was investigated using FTIR. How these bond intensities depend on fabrication parameter had also been shown.

II. EXPERIMENTAL

Boron doped P-type c-Si wafers having <100> orientation and resistivity 3-10 ohm-cm (double side polished surface) of thickness 300um were taken as starting material. Before etching, wafers were cleaned by standard cleaning procedure (solvent method and strong piranha). Silicon wafers were subjected to hot acetone treatment in ultrasonic agitation for 5 minutes followed by dipping in methanol for 2 minutes. The substrates were thereafter immersed into the piranha solution containing concentrated H_2SO_4 and H_2O_2 in 2:1 volume ratio for 15 minutes to remove the organic contaminants present in the wafer. The wafers were then dipped into 10% hydrofluoric acid (HF) to remove native oxide. Finally the wafers were subjected to standard RCA cleaning procedure. Millipore deionized water of resistivity 18 MΩ was used to rinse the samples after each step.
The cleaned silicon substrates were exposed to the vapor resulting from the reaction of Zn metal dust added one time in the solution of HF/HNO3. Samples were prepared for three different concentration of HF, HNO3 mixture (4:1, 6:1, 8:1) keeping the same etching time of 4 minute. Porosity and thickness of each porous sample were measured using gravimetric method. Table 1 listed the fabrication parameters of single etched porous samples.

Table-1

Sample name	Concentration ratio of HF, HNO_3	Time
N-1	4:1	4 min
N-2	6:1	4 min
N-3	8:1	4 min

Multiple etching was performed by exposing substrates to the vapor of two solutions of different acid concentrations periodically. A relaxation time was given between each etching step. The fabrication parameters are given in table 2.

Table-2

Sample name	Concentration ratio of HF, HNO_3	time	Relaxa-tion time	Etching step
N-4	4:1 6:1	4 min 4 min	1min	2
N-5	4:1 6:1	4 min 4 min	1 min	6

The reflectance measurement was done using shemadzu uv-vis-nir spectrophotometer (solid spec -3700). PL measurements in room temperature had been carried out by using HeAg laser of wavelength 224 nm as the excitation source, GaAs photomultiplier was used as photon detector. FTIR measurement was done using 3000 Hyperion Microscope with vertex 80 FTIR system.

III. RESULTS & DISCUSSION

A. Porosity measurement:-
Porosity of single etched porous layer was measured using gravimetric method.
%P= {(m1-m2) / (m1-m3)} x 100%

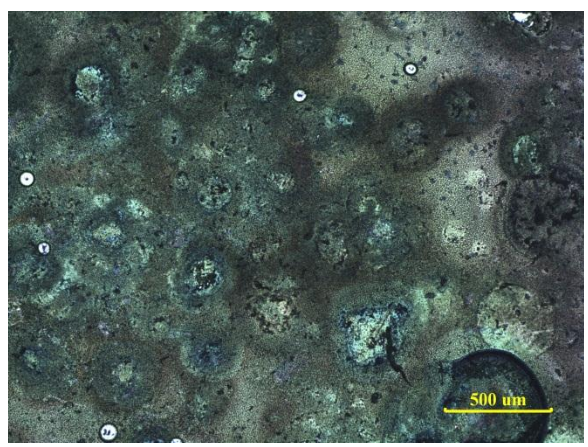
Table 3 shows porosity and thickness of porous layer for various concentration ratio of acid mixture.

Table-3

Concentration ratio of HF, HNO_3	Time	porosity	Thickness (um)
4:1	4 min	44%	1.24
6:1	4 min	50%	2.32
8:1	4 min	67%	3.48

From porosity measurement it was seen that increased concentration of HF led to higher porosity thick layer.

B. Optical microscope image:-
OM images were taken for sample N-2 and N-5 in 5X and 100X resolution.



a)

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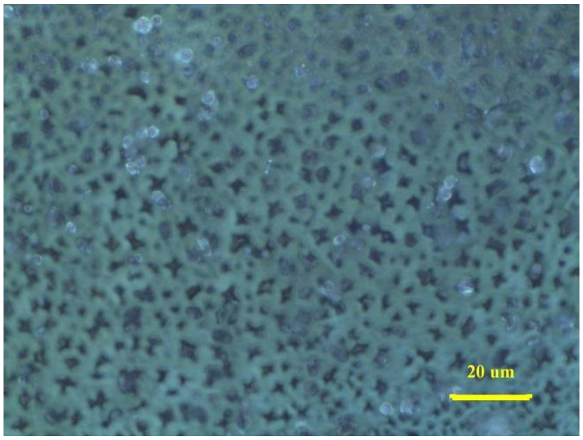

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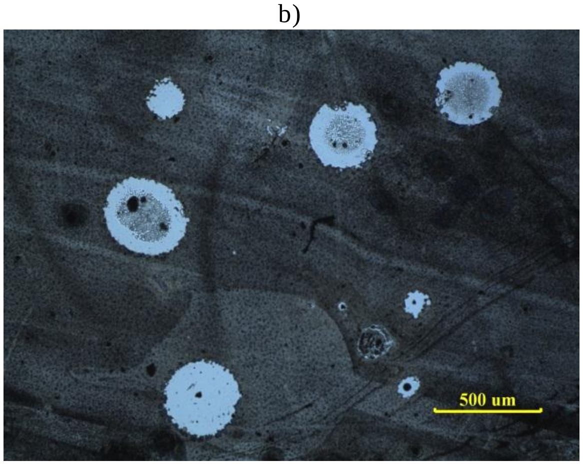

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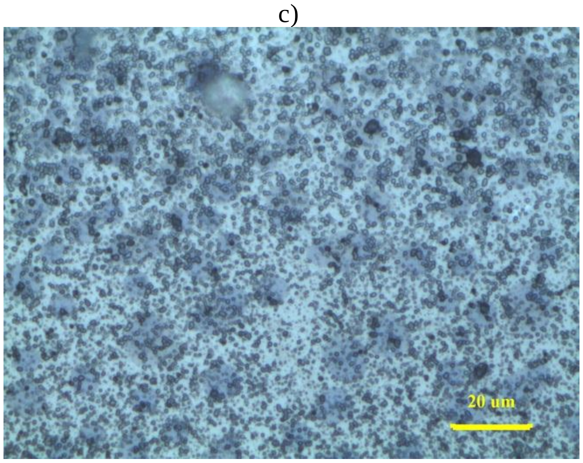

d)

Fig 1. Optical Microscope images (top view) a) sample N-2 (magn X5) b) sample N-2 (magn X100) c) sample N-5 (magn X5) d) sample N-5 (magn X100).

The Fig 1.b) shows small homogenous pores with some crystallites on the surface. Fig 1.d) shows very small pores but not homogenous. For detailed study SEM image is required. To investigate multilayer cross sectional SEM is required.

C. Reflectance Measurement:-

The Reflectance properties were investigated using Shemadzu uv-vis-nir spectrophotometer (solid spec-3700) for sample N-2, N-4, N-5.

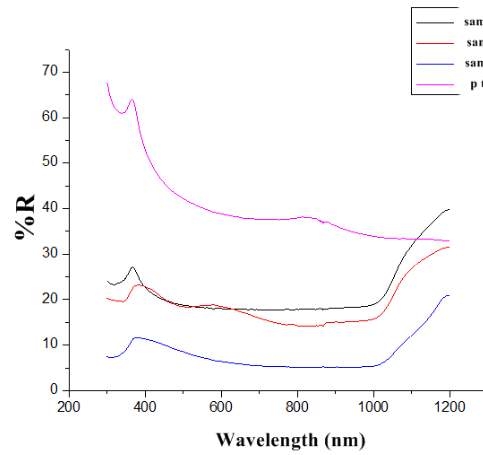

Fig. 2

From the reflectance graph it is seen that the double etched porous silicon (sample N-4) reflectance is a little lower than single etched porous structure (sample N-2). As the number of etching step is increased to six (sample N-5), the reflectance is much lower. From this reflectance study it may be concluded that the reflectance from each layer is destructive in nature. As the number of etching steps increase more layers are produced which are not periodic and overall reflectance decreases.

D. Photoluminescence:-

PL measurements in room temperature had been carried out by using HeAg laser of wavelength 224 nm as the excitation source for samples N-2 and N-5. GaAs photomultiplier was used as photon detector.

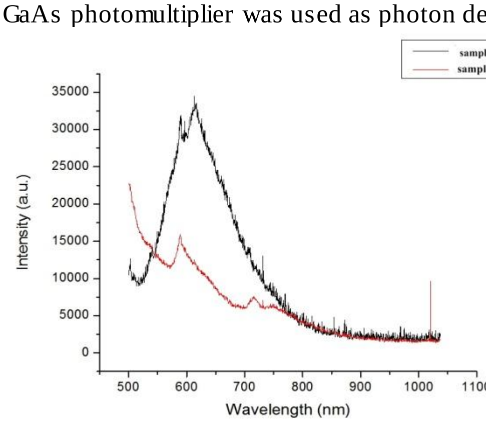

Fig. 3

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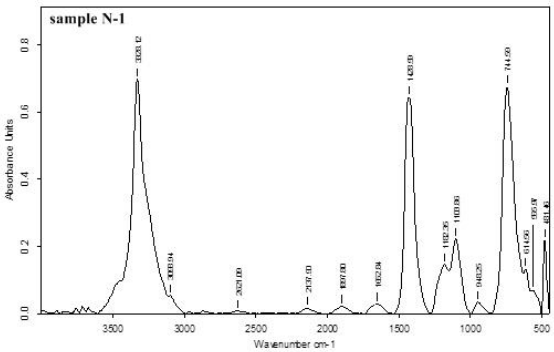
Higher intensity PL was observed for sample N-5 than N-4. It might be due to multiple etching. Further studies are required to distinguish single etched and multiple etched structure.

E. FTIR study:

The bonding structure in porous Si layers was studied using 3000 Hyperion Microscope with vertex 80 FTIR system. The readings were taken for sample N-1, N-2 and N-3 in absorption mode.



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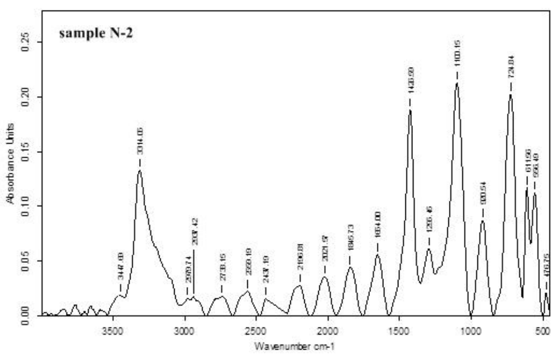

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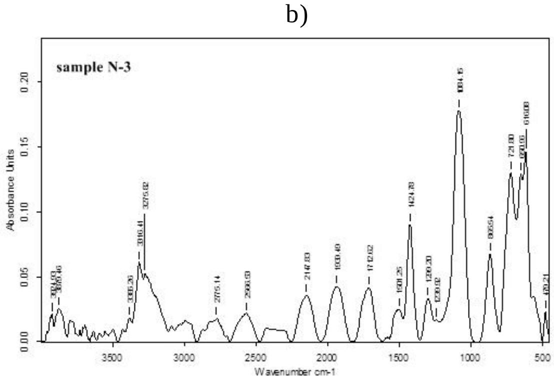

c)

Fig 4. FTIR absorption spectra of a) sample N-1, b) sample N-2, c) sample N-3.

The absorption peaks and the corresponding bonds were shown in the table 4.

Table-4

sample	Si-H bending	Si-O-H bending	Si-O-Si Asymmetric stretching	Si=O	Si-H stretching	Si-OH streching
N-1	614.56	744.59	1103.86	1428.58	2137.90	3328.12
N-2	611.56	724.84	1100.15	1426.59	2021.57,2196.81	3314.05
N-3	616.08	721.80	1084.15	1424.78	2147.83	3316.41

The FTIR analysis showed that the intensity of Si-O-Si and Si=O was decreased with increasing HF concentration. The highest peak intensity was found in sample N-1 which was etched with lowest HF concentration. Good intensity peak was also found for Si-OH bond which was not required. It might be removed by annealing after etching, which is kept for future study.

IV. CONCLUSION

Anodization is the most effective method for fabrication of multilayer porous silicon. Vapor phase etching is more simple and low cost process than anodization. No work till now has been reported to fabricate multilayer using vapor phase etching. This was an effort to make multilayer structure by multiple etching of substrates using reaction induced vapor etching. From the reflectance spectra it may be concluded that multiple etched layers are not periodic in nature so increasing etching step reflectance decreased. These structures may be used as antireflection material. FTIR study showed the dependency of bond intensity on oxidant ratio. After porous silicon formation annealing might be required to remove the Si-OH bond. The gas kinetics and rate of reaction should be controlled to optimize the multiple vapor etching procedure to produce periodic multilayer structure.

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REFERENCES

[1] L. T. Canham, "Silicon quantum wire array fabricated by electrochemical and chemical dissolution of wafers," Appl. Phys. Lett, vol. 57, Issue 10, 1990, pp. 1046–1048.

[2] S. Chan, Y.Li, L. J. Rothberg, B. L. Miller, and P. M. Fauchet, "Nanoscale silicon microcavity for biomedical sensing application" Mat. Sci. Eng. C. 15, pp.277-282 (2001)

[3] Huimin Ouyang and Philippe M. Fauchet "Biosensing using Porous Silicon Photonic Bandgap Structures" (SPIE Optics East 2005)

[4] Krisztian Kordas et al "Optical properties of porous silicon.Part II: Fabrication and investigation of multilayer structures" science direct Optical Materials 25 (2004) 257–260

[5] S. Setzu a,b, P. Ferrand a, R. Romestain, "Optical properties of multilayered porous silicon" Materials Science and Engineering B69–70 (2000) 34–42

[6] Guoguang Rong, Jarkko J. Saarinen, John E. Sipe, and Sharon M. Weiss "High Sensitivity Sensor Based on Porous Silicon Waveguide" Mater. Res. Soc. Symp. Proc. Vol. 934 © 2006 Materials Research Society

[7] Kaabi et al, " Structural and optical properties of vapor-etching based porous silicon," Cryst. Res. Technol., vol. 41, Issue 2, 2006, pp. 154–162.

[8] Saadoun et al, "Vapor etching based porous silicon: a new approach," Thin Solid Films, vol. 405, Issues 1-2, 2002, pp. 29–34.

[9] Elder A. de Vasconcelos*, E. F. da Silva, Jr., B. E. C. A. dos Santos, W. M. de Azevedo, and J. A. K. Freire "A new method for luminescent porous silicon formation: reaction-induced vapor-phase stain etch" phys. stat. sol. (a) 202, No. 8, 1539–1542 (2005)

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