International Journal
of Physics and Applications

E-ISSN: 2664-7583
P-ISSN: 2664-7575
IJOS 2024; 6(2): 136-141
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www.physicsjournal.in
Received: 05-09-2024
Accepted: 12-10-2024

Amjad H Jassim
Department of Physics, College of Sciences, University of Tikrit-Iraq

Ayman A Noori
Department of Physics, College of Sciences, University of Tikrit-Iraq

Faleh L Mater
Department of Physics, College of Sciences, University of Tikrit-Iraq

Corresponding Author:
Amjad H Jassim
Department of Physics, College of Sciences, University of Tikrit-Iraq

Investigating the Role of the annealing temperature on some properties of nanoporous porous silicon wafers

Amjad H Jassim, Ayman A Noori and Faleh L Mater

DOI: https://doi.org/10.33545/26647575.2024.v6.i2b.112

Abstract

In this research, four wafers of bulk silicon with dimensions (2x2 cm^2) were prepared. These wafers were treated with hydrofluoric acid in order to convert bulk silicon into porous silicon. This was done using the photo electrochemical method, where the slices had a resistivity of (0.02 ohm. cm) and a directionality of (110) and their majority carriers were electrons. A hydrofluoric solution was used at a concentration of (9:4) with ethanol and a lamp with a power of (500 watts). After producing the porous silicon, the samples were thermally treated at (400, 500**,** and 600) degrees Celsius for half an hour. Through the surface topographic examinations obtained from the Atomic Force Microscope (AFM), it was found that the increase in annealing temperatures reduced the porosity and decreased the thickness of the porous layer. This in turn has a direct effect if these samples are used as gas sensors or light detectors. As for the visual examination, luminescence was used to find energy gap and identifying the extent to which it is affected by thermal treatment. It was found that the energy gap increases with increasing thermal treatment temperatures**.**

Keywords: Annealing treatment, nano structure, photo electrochemical and photo luminance

Introduction

In this research, we explain the role of annealing temperature on some properties of nano porous silicon (NPS) wafers. Porous silicon (PS) is considered one of the distinctive and promising materials in the field of materials, thin films and nanotechnology due to its unique properties that allow it to be used in multiple applications, including chemical catalysis, optical detections, gas sensing, and optics [1], and this is due to its properties of structural that help it acquire these properties and characteristics [2]. Annealing or heat treatment is one of the important thermal processes that involve the structure and properties of porous silicon, inclusive pore size, cluster distribution, thickness of porous layer, electrical and optical properties [3]. This research concentrate on studying the impact of different annealing temperatures on these properties, by analyzing and comparing experimental results. The aim is to reach a deeper understanding of how to modify the properties of porous silicon to achieve the best performance in required applications. This study makes a distinguishing contribution in providing some information on how to control the properties of nano porous silicon by preventing the annealing temperature, which enhances its effectiveness in industrial and technical applications.

Theoretical part

Semiconductors, including porous silicon, can be used in various applications such as desired substrates on which materials be deposited on to produce photo detectors, sensors, and solar cells [4]. In order to know the role of annealing temperature (heat treatment) on nanoporous silicon wafers and some of its properties, this may require an in-depth review of the physical and chemical connotations and processes related to the production of (PS) and how to treat it thermally, which is called annealing. Among these notions and concepts, the term nanoporous silicon stands out, as it can be defined as a semiconductor material including of microscopic and ultra-small pores on the surface and in the crystalline structure of silicon. Porous silicon can be produced using vary techniques, like electrochemical etching, photoelectrochemical etching, photochemical etching, and laser etching. The shapes and structures of porous silicon, especially the surface morphology, may convert based on the size of the pores (micro, nano), which affects on its applications and uses, as nanoporous silicon is characterized by wide applications such as fuel cells, and applications Medical as drug delivery systems [2].

Heat treatment is the process of annealing materials to a certain temperature and then cooling them slowly for a proven period of time. This process leads to varying some of the physical and chemical properties of the treated materials, such as redistribution and recrystallization of atoms. In nanomaterials like nanoporous silicon, the annealing process plays a significant and distinctive role in causing major modifications in the internal structure of the material, which affects on its optical, structural and electrical properties. Increasing the annealing temperature usually leads to reduce and shrinking the size of the pores or redistributing them, which affects to the porosity, density of particles, optical properties and electrical conductivity of the material [5, 7]. As for the optical properties of nanoporous silicon, increasing the temperature can cause a change in the transparency and color of the porous silicon as a result of changes in the crystal structure. Based on all of the above, nanoporous silicon can be adapted for use in specific fields such as optical, gas and photoelectric sensors [8]. The internal structures of porous silicon have several shapes and patterns depending on the type of preparation and production process as well as the parameters of the bulk silicon wafer used, such as directionality, as shown in Figure (1).

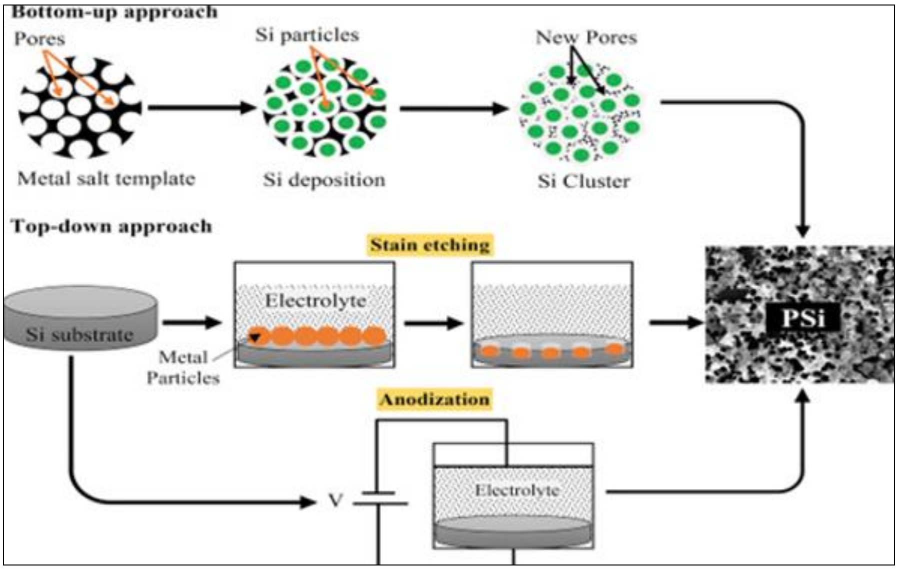


Fig 1: Schematic diagram of the production mechanism of porous silicon [9].

Experimental part

It is not possible to produce porous silicon without equations that determine the running of action and what things must be available for the process to be carried out correctly. Therefore, the equation that determines the path of action and the reaction that occurs between the surface of the bulk silicon and the acid in the presence of light is shown in equation (1) [10].

$$Si + 4HF^-_2 + h^+ \rightarrow SiF_2^-6 + 2HF + H_2 + e^- \ldots\ldots\ldots\ldots\ldots (1)$$

The mechanism of uprooting and capturing the surface silicon atoms and transporting them towards the solution in order to obtain a porous topography and a larger surface area, to make the samples more important and effective in the desired applications, then from Figure (2) we can expect what happens during the reaction.

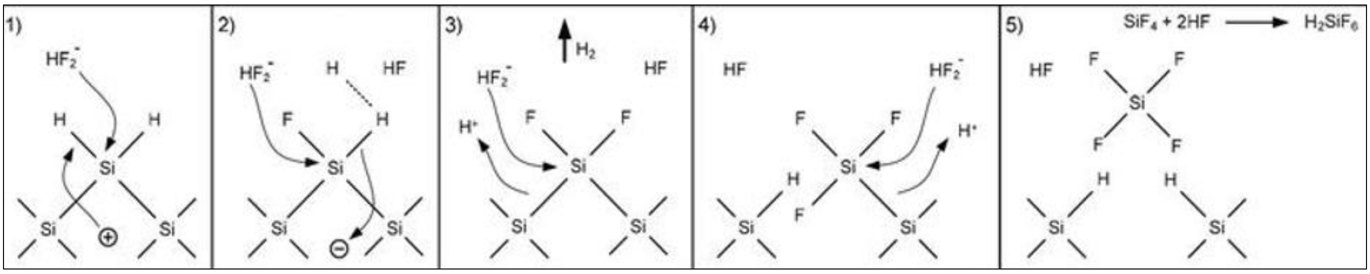


Fig 2: Divalent electrochemical dissolution of a silicon atom in hydrofluoric acid solution [8].

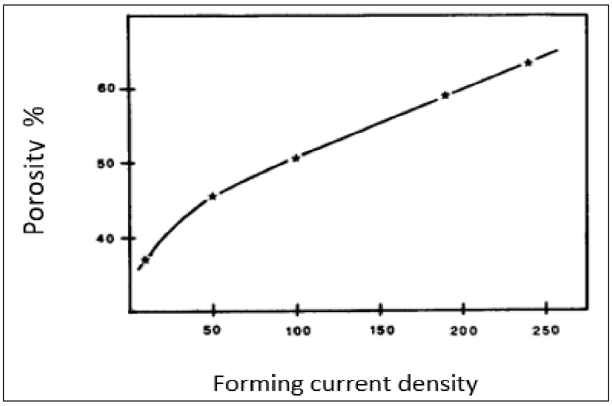


Fig 3: Gravimetric porosity as o function of forming current density for porous layers prepared on heavily doped N-type silicon in 25% HF [8].

As mentioned previously, the photo-electrochemical etching method was used because we used silicon slices with a majority of negative carriers. Therefore, light must be used to generate enough gaps to carry out the etching process and remove some of the surface of the sample, as demonstrated in the figure below.

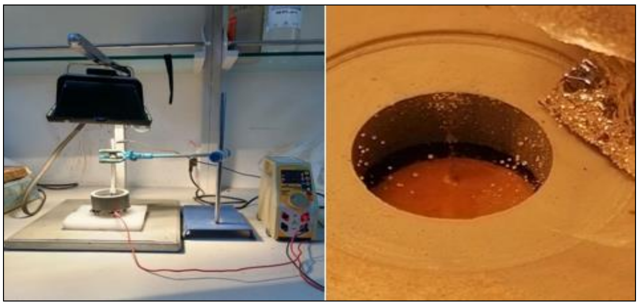

A B

Fig 4: (A) Exhibit the photo electrochemical etching system (B) shows the wafer during etching

Figure (4) includes the system used in the etching process and the shape of the sample during the etching process, showing the color of the sample and the bubbles resulting from the reaction that result from etching operations on the surface of the sample [11], due to the flow and penetration of hydrofluoric acid into the silicon structure that makes up the sample.

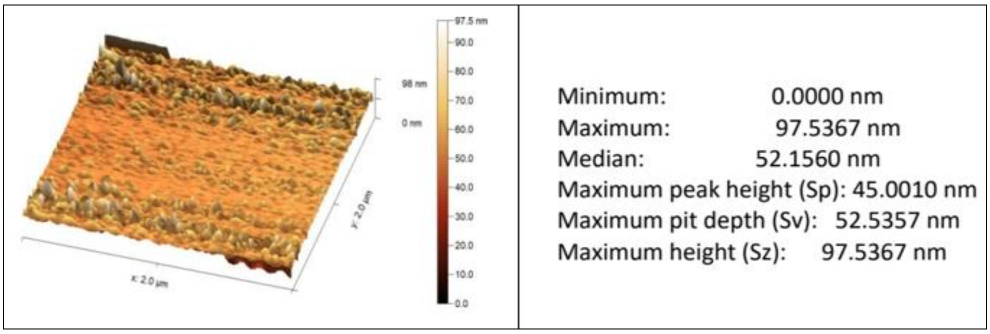
Results and Discussion

In this part of the research, several parameters and variables were discussed that were applied to bulk silicon slices, through which porous silicon was produced. Several tests were used to determine the properties and qualities of the produced slices and among those tests were;

A. Morphological part

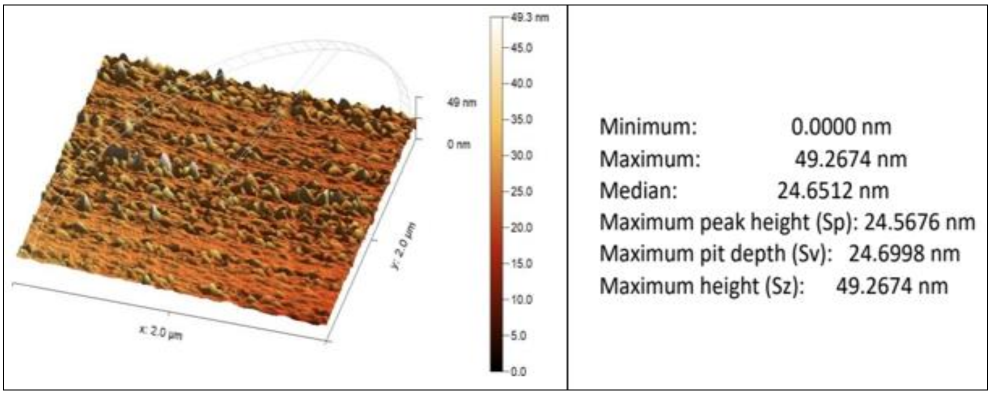
(1) AFM examination

Through this examination, the surface topography of the produced slices was known.



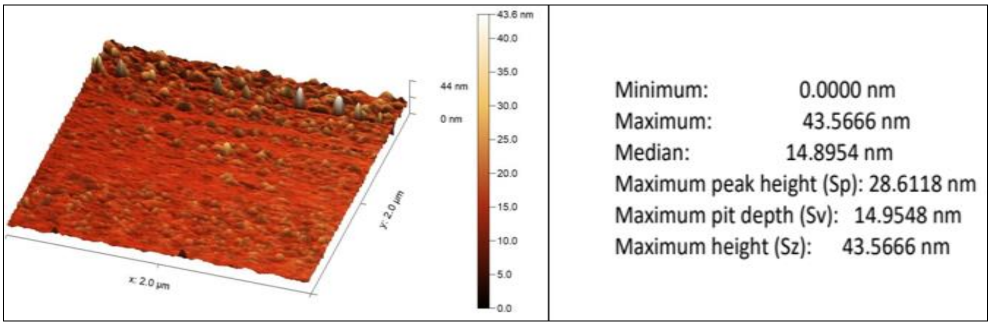
Minimum: 0.0000 nm
Maximum: 97.5367 nm
Median: 52.1560 nm
Maximum peak height (Sp): 45.0010 nm
Maximum pit depth (Sv): 52.5357 nm
Maximum height (Sz): 97.5367 nm

Fig 5: Image of AFM for PS prepared by PEC etching technique with a constant time of (7 min), and constant current density (15 mA /cm2) with ethanol: HF mixing (4:9) with thermally treated at (400) °C.



Minimum: 0.0000 nm
Maximum: 49.2674 nm
Median: 24.6512 nm
Maximum peak height (Sp): 24.5676 nm
Maximum pit depth (Sv): 24.6998 nm
Maximum height (Sz): 49.2674 nm

Fig 6: Image of AFM for PS prepared by PEC etching technique with a constant time of (7 min) and constant current density (15 mA /cm^2) with ethanol: HF mixing (4:9) with thermally treated at (500) °C.



Minimum: 0.0000 nm
Maximum: 43.5666 nm
Median: 14.8954 nm
Maximum peak height (Sp): 28.6118 nm
Maximum pit depth (Sv): 14.9548 nm
Maximum height (Sz): 43.5666 nm

Fig 7: Image of AFM for PS prepared by PEC etching technique with a constant time of (7 min), and constant current density (15 mA /cm^2) with ethanol: HF mixing (4:9) with thermally treated at (600) °C

Table 1: The calculated topography characteristics of PS samples PEC etching technique with a constant time of (7 min), and constant current density (15 mA /cm2) with ethanol: HF mixing (4:9) with thermally treated at (600) °C

Thermally treated at °C	RMS roughness (Sq) nm	Mean roughness (Sa) nm
400	6.2393	4.335
500	3.547	2.648
600	2.68935	1.9675

Through the figures (5),(6) and (7) in addition to the table (1) Its find that by increasing the annealing temperature, the thickness of the porous layer decreased, and this appeared noticeably upon thermal exposure to the second eye, as the thickness of the porous layer decreased to approximately half. This indicates that the annealing process directly affected the Surface compositions and the expected dissolution occurred, which would reduce unwanted cracks within the sample.

Annealing of nanoporous silicon wafers leads to a decrease in grain size as a result of changes in the crystalline structure of silicon. During the annealing process, the material is heated to a certain temperature and then slowly cooled, allowing the atoms to rearrange within the crystal lattice.

In nanoporous silicon wafers, the annealing process results in the growth of large crystalline grains at the expense of small ones. This process is known as "Ostwald Ripening", where large grains increase in size while small grains shrink or disappear due to the migration of atoms towards larger grains that are more energetically stable, exactly as happened in the examined samples used in this research, and the roughness of the sample surfaces was decrease with increasing the annealing temperatures and this agreement with [12].

Cross-section examination

through this examination, the thickness of the actual pores produced by photo electrochemical drilling and scratching was found, and it was found that the largest thickness of the porous layer produced was at an annealing temperature of (400), This is because as the temperature increased, the pores began to disappear due to the melting of the upper terrain, which resembles spikes or plateaus, and gradually transformed into something resembling plains or flat surfaces. Therefore, the thickness of the porous layer decreased with the increase in the annealing temperature. This was also reflected in the percentage of particles forming on the surface, and this is agreement with [13].

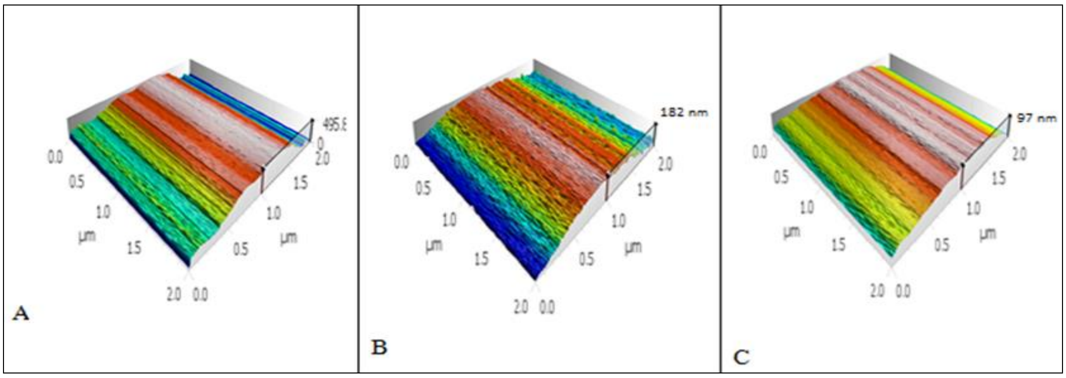
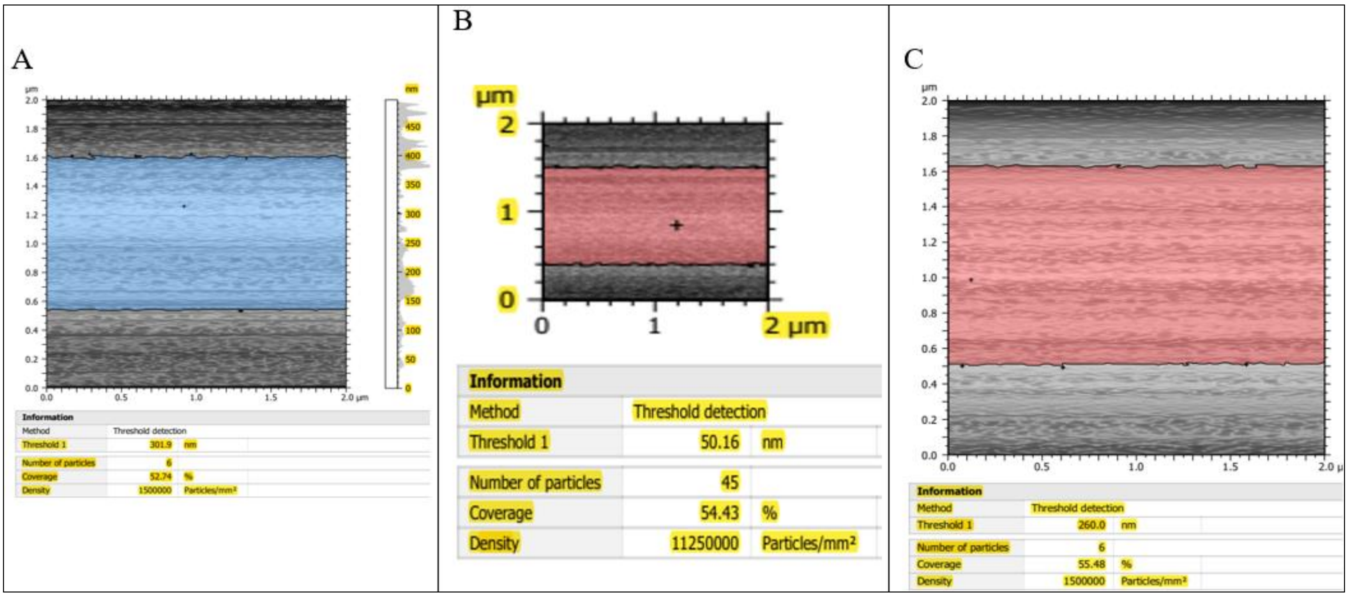


Fig 8: Images of Cross-section for PS wafers prepared by PEC etching technique with a constant time of (7 min), and constant current density (15 mA /cm^2) with ethanol: HF mixing (4:9) with thermally treated at (A=400, B = 500, C= 600) °C

Density of Particles

Looking closely at Fig.(8), and table (2), Its find that the number of particles formed on the surface of the samples increases significantly as a result of the fragmentation of the large particles and the formation of two smaller particles, which increases the number of particles measured on the surface. This is agreement to some extent with the fluorescence results; in addition to that the sizes of the valleys between the particles produced on the surface also decrease with increasing degree. The heat of annealing is due to the fusion of large particles and their transformation into smaller particles, which reduces the valley distances produced between them.



A

Information		
Method	Threshold detection	
Threshold 1	301.9	nm
Number of particles	6	
Coverage	52.74	%
Density	1500000	Particles/mm²

B

Information		
Method	Threshold detection	
Threshold 1	50.16	nm
Number of particles	45	
Coverage	54.43	%
Density	11250000	Particles/mm²

C

Information		
Method	Threshold detection	
Threshold 1	260.0	nm
Number of particles	6	
Coverage	55.48	%
Density	1500000	Particles/mm²

Fig 9: Images of PS wafers and its density of particles which prepared by PEC etching technique with a constant time of (7 min), and constant current density (15 mA /cm^2) with ethanol: HF mixing (4:9) with thermally treated at (A=400, B = 500, C= 600) °C

Table 2: The calculated for density of particles PS samples which prepared by PEC etching technique with a constant time of (7 min), and constant current density (15 mA /cm^2) with ethanol: HF mixing (4:9) with thermally treated at (A=400, B= 500, C=600) °C

thermally treated at °C	Density (Particles/mm²)	Valley depth (Svkx) nm
400	$15*10^5$	164.4
500	$112*10^5$	144
600	$15*10^5$	29

Through paragraphs 1, 2, and 3, we find that the annealing process reduced the structures produced on the surface, and this in turn leads us closer to the results of quantitative confinement because the sizes are possible microscopically to approach (10) nanometers, and this may produce new and important specifications for the parent material before it is etched and treated thermally, in addition to treating the materials thermally. It may remove the internal stresses that contribute to making the produced material more ideal by using it in other important applications such as gas sensors or use them as anti-reflective bases [14].

B. Optical part

1. Energy Gap examination

For the purpose of finding the energy value of the produced samples, which are porous silicon samples, and the extent to which the energy gap is affected by etching processes, we resort to the fluorescence method. By plotting between wavelength and intensity and from the highest peak, we can find the energy gap through the equation E=hc/λ as in the figures below:

Through figures (10, 11, and 12) it found that the energy gap increased with the increase in annealing temperatures. This is due to the increase in temperature leading to the melting of the particles formed on the surface, which are relatively large compared to the particles that were produced or appeared with higher annealing temperatures. This led to an increase in the energy gap because as the particle size decreases. The energy gap increases due to the quantum confinement effect that dominates the behavior of the nanomaterial, especially the annealed porous silicon, and this is consistent with [15].

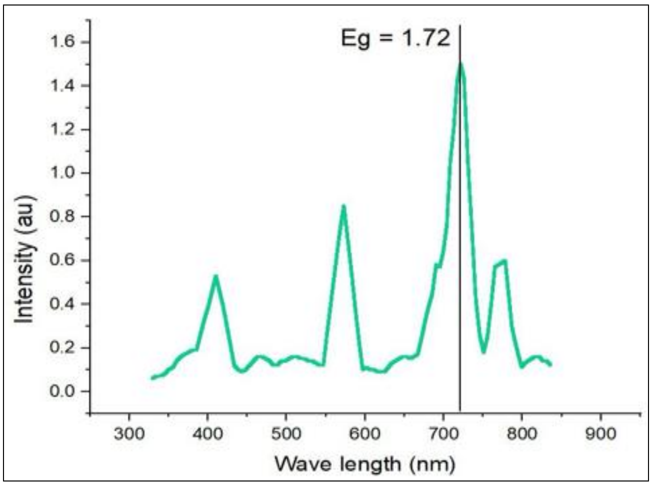


Fig 10: Photo luminesce Image of PS wafer which prepared by PEC etching technique with a constant time of (7 min), and constant current density (15 mA /cm^2) with ethanol: HF mixing (4:9) with thermally treated at (400) °C

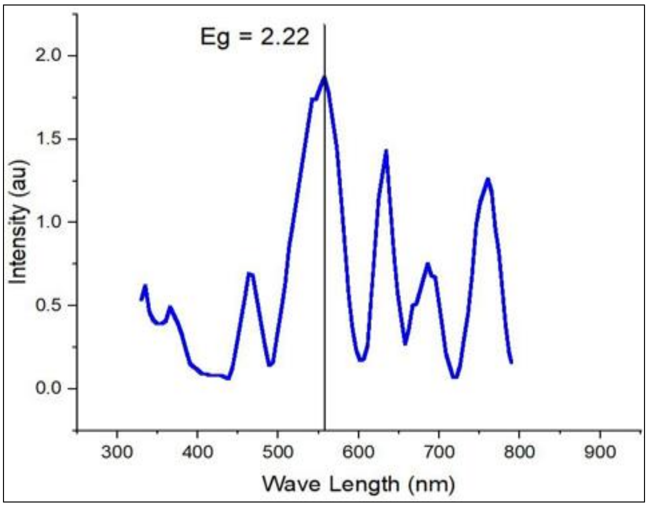


Fig 11: photo luminesce Image of PS wafer which prepared by PEC etching technique with a constant time of (7 min), and constant current density (15 mA /cm^2) with ethanol: HF mixing (4:9) with thermally treated at (500) °C

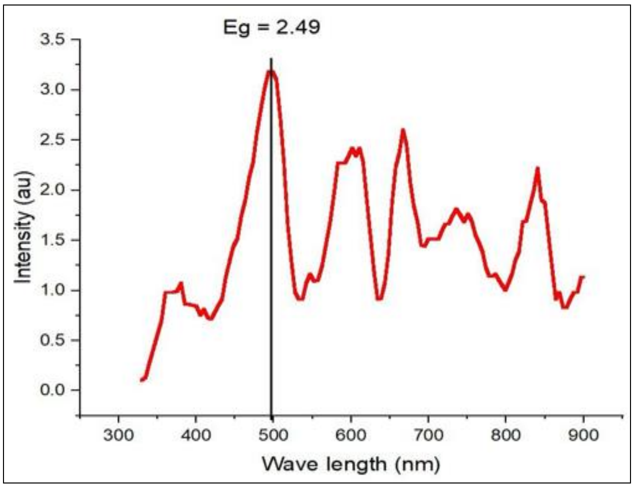


Fig 12: photo luminesce Image of PS wafer which prepared by PEC etching technique with a constant time of (7 min), and constant current density (15 mA /cm^2) with ethanol: HF mixing (4:9) with thermally treated at (600) °C

Conclusions

What was concluded in this research is that the surface topography of the produced samples was significantly and clearly affected by the heat treatment. As the surface roughness and the shapes of the structures formed on the surface changed, as the surface roughness decreased with the increase in the annealing temperature, which caused the melting of the formed peaks, which in turn was reflected in the density of the surface particles, as the number of particles increased with the increase in the annealing temperature. For the energy gap, it increased with the increase in the annealing temperature because the grain size decreased as a result of the formation of small clusters and the appearance of gaps and pores approaching 10 nanometers, which caused the dominance of the effect of quantitative confinement

References

1. Nayel HH, Al-Jumaili HS. Fabrication of highly sensitive NH3 sensor based on mixed In2O$_3$ – AgxO nanostructural thin films deposited on porous silicon. J Univ Anbar Pure Sci. 2019;13(2):40-47. DOI:10.37652/juaps.2022.172118.
2. Al-Jumaili HS, Jasim MN. Preparation and characterization of ZnO: SnO$_2$ nanocomposite thin films on porous silicon as H$_2$S gas sensor. J Ovonic Res. 2019;15:81-7. Available from: https://www.researchgate.net/publication/331732052.
3. Benyahia B. Thermal annealing dependence of some optical properties of plasma-modified porous silicon. Appl Surf Sci. 2010;257:1105-11. Available from: https://www.researchgate.net/publication/369794775.
4. Najim JA, Alwahid RA, Rasheed HK. Study the surface topography and electrical properties of GaAs: In / c-Si composite thin film. J Univ Anbar Pure Sci. 2018;12(1):88-97. DOI:10.37652/juaps.2022.171604.
5. Canham LT. Silicon quantum wire array fabrication by electrochemical and chemical dissolution of wafers. Appl Phys Lett. 1990;57(10):1046-8. DOI:10.1063/1.103561.
6. Cullis AG, Canham LT, Calcott PDJ. The structural and luminescence properties of porous silicon. J Appl Phys. 1997;82(3):909-65. DOI:10.1063/1.366536.
7. Jassim AH, Al-Samarai AE, Ibrahim IM. Study the performance of some rare earth UV detectors. Solid State Technol. 2022;65:91-100. Available from: https://www.researchgate.net/publication/360521285.
8. Herino R, Bomchil G, Barla K, Bertrand C, Ginoux JL. Porosity and pore size distributions of porous silicon layers. J Electrochem Soc. 1987;134(8):1994-2000. Available from: https://iopscience.iop.org/article/10.1149/1.2100805.
9. Smith RL, Collins SD. Porous silicon formation mechanisms. J Appl Phys. 1992;71(8):R1-22. DOI:10.1063/1.350839.
10. Geppert T, Schweizer S, Gösele U, *et al*. Deep trench etching in macroporous silicon. Appl Phys A. 2006;84:237-42. DOI:10.1007/s00339-006-3628-7.
11. Lehmann V. Electrochemistry of silicon: Instrumentation, science, materials and applications. Germany: Wiley-VCH; c2002. Available from: https://www.smbstcollege.com.
12. Farshid K. Porous silicon, porosity - process, technologies and applications. Science and Technology Park, University of Tehran, Tehran, Iran. 2018;1:9. Available from: https://www.intechopen.com/books/6175.
13. Sheng L. Effects of annealing treatment on microstructure and tensile behavior of the Mg-Zn-Y-Nd alloy. J Magnes Alloys. 2020;8(3):601-613. DOI:10.1016/j.jma.2019.07.011.
14. Noor M. Study the properties of porous silicon for P-type and N-type bulk silicon. Thesis, College of Education for Pure Sciences, University of Kerbala; c2021.
15. Jin Z. Optical properties and spectroscopy of nanomaterials. University of California, USA; 2009. Available from: https://books.google.iq/books?id=AfLFCgAAQBAJ&lpg=PR7&ots=WXJlC-BZC-&dq=Optical%20properties%20of%20Nanomaterials%20porous%20silicon&lr&hl.