



A novel method for calculating electrical and thermal parameters of graphene nanoparticles on a porous silicon prepared by drop casting method

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Due to its prospective use in chemical sensors, flat-panel displays, and optoelectronics, porous silicon (PS) has recently garnered considerable scientific interest. (PECE) Photo-Electrochemical Etching is used on silicon wafer (Si) (N-type) with 20% hydrofluoric acid (HF) and ethanol (C₂H₅OH) concentration at 15 mA/cm² current density for 15 minutes, nanostructure porous silicon (PS) is fabricated. Drop casting method has been used to deposit the graphene on the n-type Si substrate. Several techniques are used to characterize the porous silicon nanostructures that are manufactured. The production of porous silicon is confirmed by X-ray diffraction (XRD), and the crystal size decreases in the direction of the face-centered cubic structure's nonmetric scale. The Scanning Electron Microscope (SEM) reveals that the structure of PS with orientation (100) resembles a sponge. The composition of the samples is depicted using (EDXA) energy dispersive X-ray Spectroscopy. (FTIR) Fourier-transform infrared spectroscopy has been used to measure the bonds of the materials prepared. Atomic force microscope is utilized to calculate the roughness of the samples. UV-vis spectroscopy is utilized to calculate the optical properties of the samples such as; transmittance; reflectance; and refractive index. Drop casting method has been used to deposit the graphene on the n-type Si substrate. The electrical behavior of the prepared samples has been examined; in addition, the thermal behavior of the samples is examined.

Keywords: Porous silicon (PS); Photo-electrochemical etching (PECE); Hydrofluoric acid (HF); Ethanol (C₂H₅OH); Electrode.

1. INTRODUCTION

Because of the properties of nanomaterials, including silicon nanostructures, which include their ease of access, low cost, low toxicity, and complete compatibility, this has led to them playing an important role in the nanostructures of various materials. One of the most important features of silicon and semiconductors is that when dimensions are reduced, wonderful changes occur in the optical, electrical, and electronic mechanical properties of the devices. Which relates to these materials, in addition to the fact that the movement of electrons in these materials is very high. A complex network of nanometer-scale pores divided by thin walls is known as porous silicon (PS). Based on the dimensions and widths of the pores, the porous structure is divided into three categories: macroporous, mesoporous, and nonporous [1]. Previously, nanostructures had many applications, such as in the field of sensors, biophotonics, and optoelectronics. After the change in the surface properties and dimensions (morphology) of the porous silicon, this change affected the accuracy of the devices or applications. The physical properties of the porous silicon varied according to the current, etching time, and concentration of Hydrofluoric acid (HF) in addition to the use of other types of stimulants [2]. Since Canham's discovery of nonporous silicon's ability to emit light in the visible spectrum, porous silicon has attracted a lot of attention [3]. Therefore, applications and devices have diversified in many fields due to the tremendous properties of silicon, such as gas sensors, solar cells, biosensors, and electrical and optical devices. All of this development is due to the low cost and ease of access to these structures. [4]. In 1990, porous silicon is discovered through the process of electropolishing silicon using diluted hydrofluoric acid. It is found that it has a nanostructure with porous holes inside it. After several years, it is found that PS possesses an important property, namely, visible luminescence. Studies have shown that PS has electrochemical and optical conductivity. Many different methods have been used to fabricate nanostructured PS such as chemical and electrochemical etching. The electrochemical etching process is the method used to create PS on a silicon wafer in an HF solution, especially in electroluminescence device applications. The method of electrical drilling without using external bias is one of the successful methods in forming PS and is less expensive. Numerous methods have been explored and investigated, but the optical quality of this material depends not only on porosity but also on the structure in which the porous silicon is generated. Although the features of porous silicon have been explained by the quantum confinement model, some evidence indicates that the luminescence qualities may cause by more than just quantum confinement effect [5]. PS has structures of crystals in its voids, and these voids create a shape for PS that resembles a sponge with pores. The channels surrounding the porosity resemble crystalline skeletons, so electronic circuits rely on PS layers, which are carefully developed to study electrical properties. Utilizing the semiconductor-focused photo electrochemical etching (PECE) process, distinctive structures in electric and photonic devices are created, such as grating in laser devices and integrated lenses on light-emitting diodes [6]. In the current work, we created PS for low resistivity n-type silicon using the photoelectrochemical etching approach and graphene with porous silicon (GNPs:PS) prepared by drop casting method has been prepared; in addition; the effect of etching current density on their morphological and structural properties have been analyzed.

2. EXPERIMENTAL WORK

Porous silicon (PS) layer is generated utilizing monocrystalline Si wafers n-type, with (1.5-4) $\Omega\cdot\text{cm}$ resistivity and (100)-orientation. Si wafer clean by used ethanol and alcohol using ultrasonic bath for few minutes. For photo-electrochemical etching (PECE) of Si wafers, A 15 min etching period and a 15 mA/cm^2 current density are used in the setup of the etching procedure. The solution contained a mixture of 20% hydrofluoric (HF) and purity ethanol ($\text{C}_2\text{H}_5\text{OH}$) at 1:1 ratio (by volume) filled in Teflon cell. The experimental set-up of PECE method which can be used to anodize Si material and drop casting method is illustrated in Figure 1. The sample is illuminated with a laser beam. The sample

are cleaned with ultrasonically treated distilled water after the development of porous structures on Si wafer, and then they are dried in a hot air stream. Grapheme material is deposited by droplet method (five drops) on PS surface.

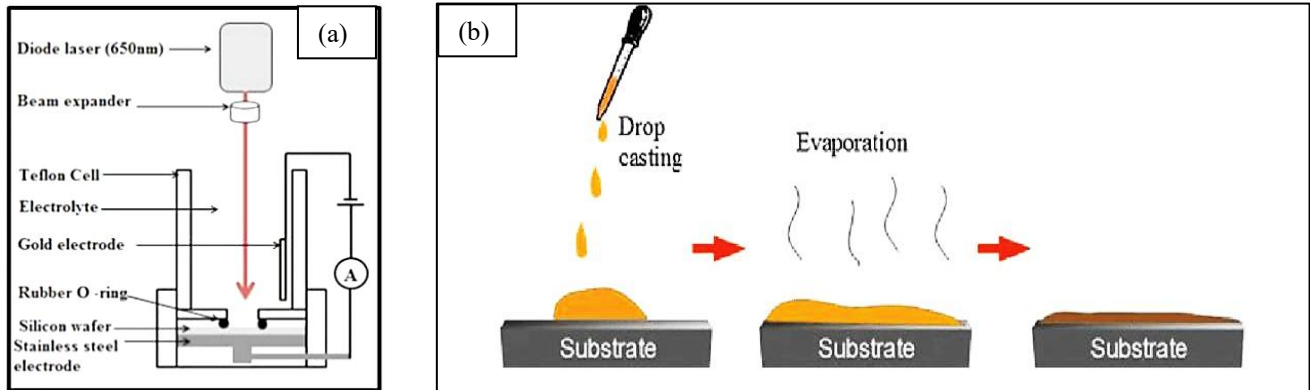


Figure 1 (a) photo-electrochemical etching (PECE) method of Si wafers, (b) Drop casting method.

3. MATERIAL AND METHODS

Materials such as hydrofluoric (HF), Si wafers (100) n-type, ethanol (C_2H_5OH), distilled water, alcohol, graphene nanopowder with average particle size 12 nm have been used in this paper, while, the devices such as X-Ray diffraction type (SHIMADZU-6000) from Japan, Scanning electron microscope (SEM) type (FEI© Inspect F50) from USA, Energy Dispersive Spectrometer (EDXA), model (AxiaChemi SEM system) from USA, Fourier transform infrared spectroscopy (FTIR); model SHIMADZU-1800 from Japan, Atomic force microscopy (AFM), model NaioAFM 2022, Nanosurf Switzerland) and UV-Visible spectroscopy model (SP-8001 Shimadzu) from Taiwan, ultrasonic bath, model (BRANSON Ultrasonic-2 CAMDA 19 spc), gold mesh (Local manufacture) have been used to do the necessary measurements in this paper. Results and Discussion.

4. RESULTS AND DISCUSSION

4.1. XRD of PS

Figure 2 A and B depict the diffraction patterns of a PS sample fabricated by (PECE) at a current density of 15 mA/cm^2 for 15 minutes and a GNPs:PS sample prepared by Drop Casting Method (DCM) at a fixed current density of 15 mA/cm^2 for 15 minutes, respectively. Specific angular positions of the x-ray beam with regard to the incident beam are determined by the phases of the sample. When crystal size approaches the nanometric scale, diffraction peaks broaden, and the width of the peak is directly proportional to the size of the nanocrystalline domains. It is common knowledge that crystallite size by calculating (FWHM) the full width at half maximum of a diffraction pattern and applying the Scherrer equation [7]

$$D_p = K \lambda / (\beta \cos \theta) \quad (1)$$

where D_p = Average Crystallite size; β = Line broadening (rad); K = Scherrer constant $K = 0.95$; θ = diffraction (Bragg) angle; λ = X-Ray wavelength. Estimated using the Scherrer equation, Table 1 displays the pore sizes derived from PS samples. PS XRD spectra revealed a broadened peak at $2\theta = 33.05^\circ$ with (211) plane, confirming the formation of fissures on the surface of crystalline silicon. This peak's presence in PS structures confirms that the cubic structure of crystalline silicon is maintained even after the formation of pores and presence of GNPs [8]. Other peaks are observed at $2\theta = 26.6$,

33.05, 47.36, 56.17 and 61.77° Figure 2-b. The structure and lattice parameters of graphene film deposited by the drop casting method on a PS substrate are analyzed using a LabX XRD 6000 SHIMADZU XR – Diffractometer equipped with Cu K- radiation ($\lambda=0.154059$ nm, voltage 30 kV, current 15 mA, scanning speed = 4 °/min). Figure 2 shows the spectra of diffraction patterns are obtained by varying 2θ from 0° to 65°. The X-ray diffraction spectrum of a thin film has two distinct peaks. It shows that the film has crystal planes and is polycrystalline, (002) suggesting that graphite's carbon structure is highly organized as shown in Table 2, and (211) as a result of PS. In addition, using Equation 1, the average particle size of PS is 72.5 nm (Table 1), while for GNPs is calculated to be approximately 43.5 nm [red values] (Table 2). In addition, lattice strain (ϵ) in (nm) and dislocation density (δ) in (nm) have been calculated from XRD pattern for all the obtained peaks and given in Table 1 and 2.

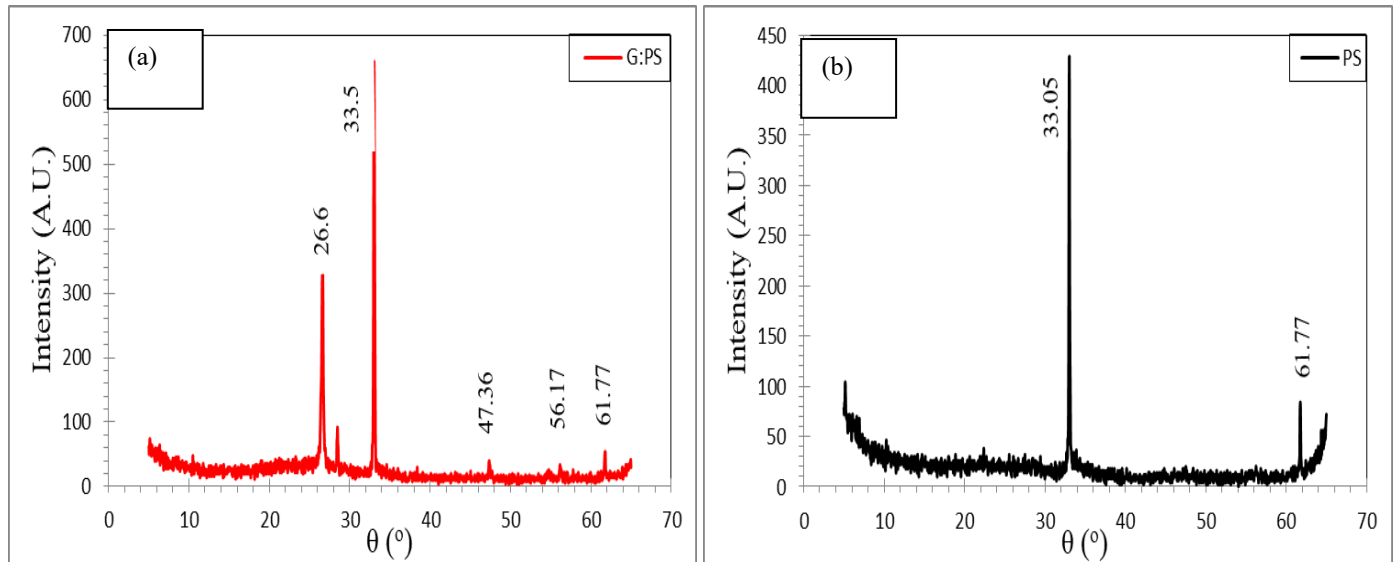


Figure 2 XRD diffraction of (a) PS, (b) GNPs:PS.

Table 1 An overview of PS nanostructure's XRD characterization Figure 2 A.

2theta (°)	theta (°)	d(A°)	β (°)	D (nm)	δ (nm)	ϵ (nm)
33.0538	16.52694	2.70789	0.11021	72.4	1.786705563	4.631586013
61.7782	30.88910	1.50045	0.12250	66.9	1.764724419	4.603007543

Table 2 Summary of XRD characterization for GNPs:PS nanostructure Figure 2 B.

2theta (°)	theta (°)	d(A°)	β (°)	D (nm)	δ (nm)	ϵ (nm)
26.6285	13.3142	3.34490	0.23050	34.5	8.056596	9.835106
33.0558	16.5290	2.70773	0.16450	48.5	3.981230	6.913722
47.3650	23.6825	1.91775	0.16450	49.1	3.629305	6.60108
56.1790	28.0895	1.63598	0.17330	47.0	3.734952	6.696468
61.7721	30.88605	1.50059	0.15460	53.0	2.810941	5.809371

4.2. SEM

Scanning electron microscopy is a useful technique for studying the morphology of materials. Since the surface morphology of a film plays an essential role in numerous applications, the morphology of the film should be of high quality [9]. As shown in the figure, it possesses large circular pores and some of these pores are interconnected with arbitrarily oriented structures with diameters ranging from 163 nm to 0.5 nm. Figure 3 A and B depicts a SEM image of GNPs sheets that appear as thin sheets with distinct boundaries, wrinkling, and folding. The average sheet thickness is approximately (1020) nm [10]. Figure 3 C and D depict a graphene NPs deposited on PS. It is observed that graphene sheets are disseminated on the PS substrate's surface.

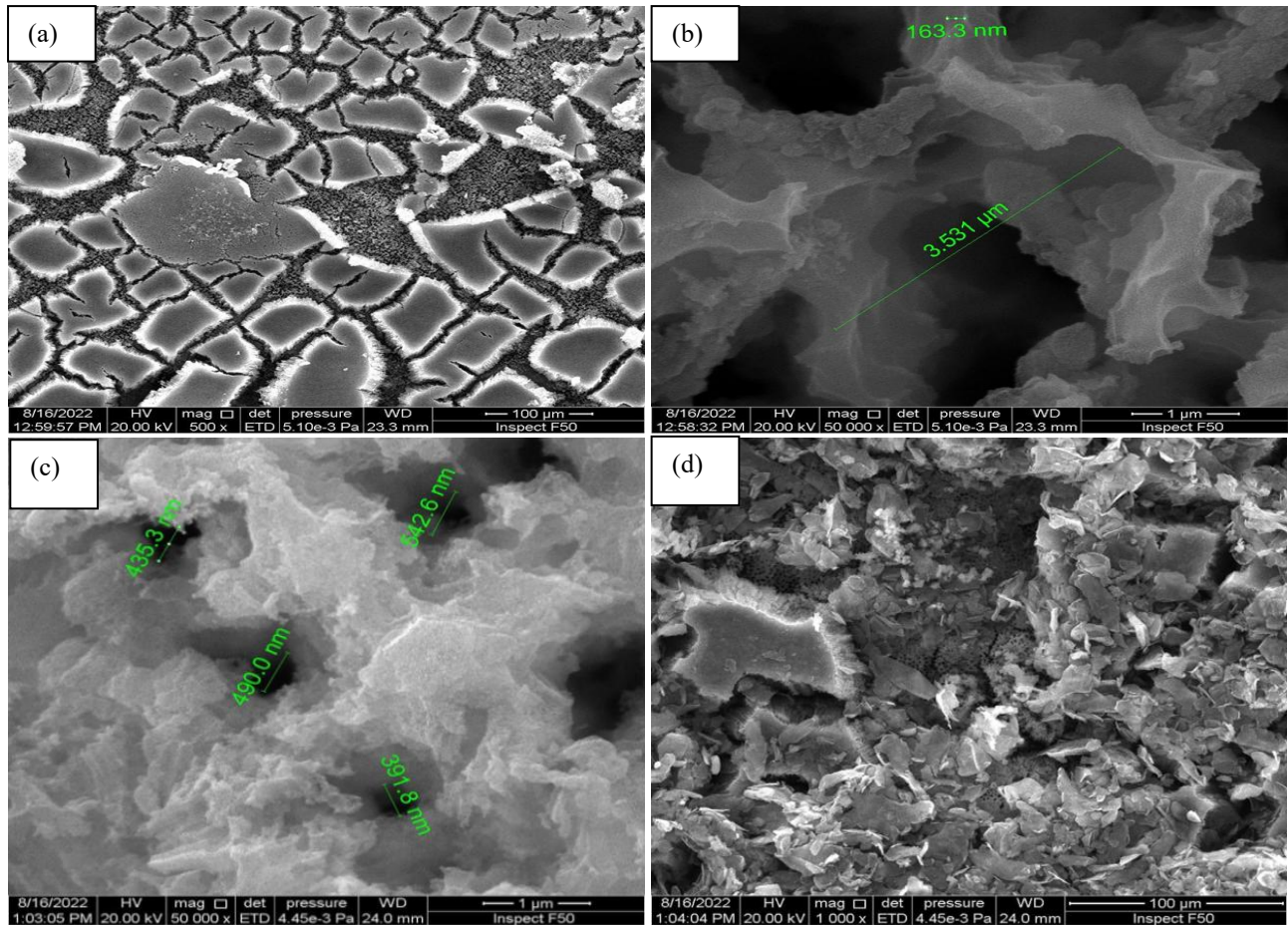


Figure 3 (a) and (b) Porous silicon is using (PECE) method, (c) and (d) GNPs:PS using drop casting method at a room temperature.

4.3. EDXA

EDXA analysis utilizes X-ray spectrometers. EDXA is a widely employed analytical method for detecting the presence and quantity of chemical elements in a sample. In conjunction with scanning electron microscopy (SEM), it is utilized. When the sample is bombarded by the SEM's electron beam, the sample's atoms emit electrons. The resulting electronic vacancies are supplied by electrons in a higher energy level, which cause X-ray to be released in order to balance the energy differential between the two electron states. The relaxation process during X-ray emission is a unique property of each element. Figure 4 shows the prepared film of the EDXA spectrum. These emissions (spectra) indicate the presence of Si, O, and C elements only, and therefore these spectra indicate the purity of

the prepared films. The Si element appears due to the use of a semiconductor silicon wafer as a substrate, and the presence of O due to the use of water as a solvent, carbon in a greater percentage (dissolved) 66%, the percentage of oxygen 18.6%, and silicon 14.4%. All composition of film discovered by EDXA is displayed in Table 3.

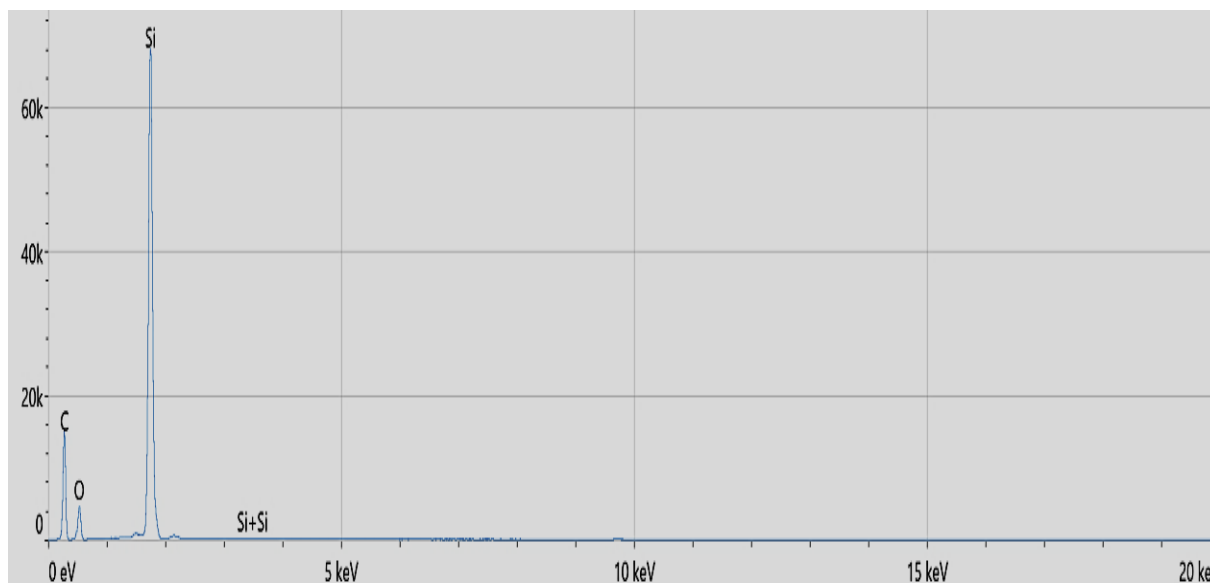


Figure 4 EDXA pattern for GNPs:PS.

Table 3 Illustrates the composition of film determined by EDXA technique.

Elements	Atomic weight (%)	Weight (%)
C	66.6	52.8
O	18.6	19.7
Si	14.8	27.5

4.4. FTIR analysis

Figure 5 shows the graphene and graphene with water FTIR spectra. The existence of oxygenated functional groups resulting from the successful oxidation of graphene accounts for a few distinct distinctive peaks in GNPs. Table 4 show the broad adsorption band.

Table 4 Illustrates the functional group of the prepared samples via FTIR.

Functional group	Wavenumber(cm^{-1})	Description
O-H	3439	Stretching vibrations
C=O (carboxyl)	1678	Stretching vibrations
C=C (carbonyl)	1639	Stretching vibrations
C-O (carboxyl)	1377	Stretching vibrations
C-O (alkoxy)	1095	Stretching vibrations

Figure 5 depicts the FTIR spectra of GNPs and hybrid nanocomposites with various loading ratios. The appearance of a peak between 500 and 600 cm^{-1} distinguishes GO from the hybrid nanocomposite in which the peak indicated the presence of graphene-O. The maximal intensity of the hybrids is found to be very similar, with the exception of the water/graphene 3:1 hybrid, which likely exhibited a higher loading ratio of grapheme [11].

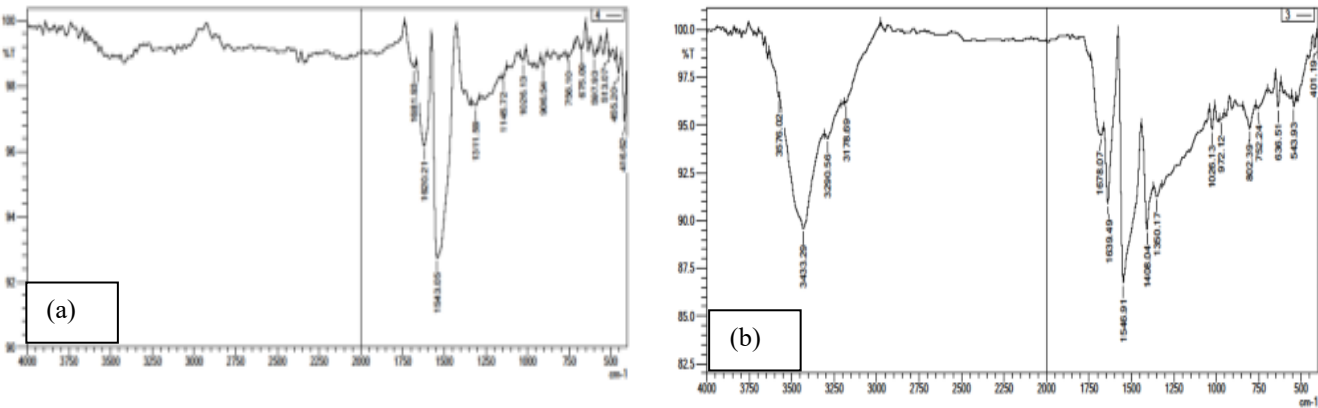


Figure 5 FTIR for (a) graphene pure, (b) GNPs (0.6 M=molarity) with H₂O 60%.

4.5. AFM analysis

Figure 6 depicts the outcome of AFM, an image of AFM conducted on a nanometric scale. The average roughness (R_{ave}) and grain size (D_{ave}) are listed in Table 5 for PS and GNPs:PS. At a current density of $J=15 \text{ mA/cm}^2$ for 10 minutes, the average PS diameter is 65.15 nm, while the GNPs:PS sample averaged 105.8nm. Figure 6 (A and B) reveal that the surface layer of PS has a sponge-like structure with a high density and uniformity of pores, while Figure 6 (C and D) depict the deposition of GNPs on the surface of PS [12]. This figure demonstrates that the colloids of GNPs: PS fills the pores, resulting in an increase in pore size and a change in surface roughness, as shown in Table 5.

Table 5 The AFM results for PS and GNPs:PS at room temperature by DCM.

Sample	D_{ave} (nm)	R_{ave} (nm)
PS	72.5	105.8
GNPs:PS	43.5	65.15

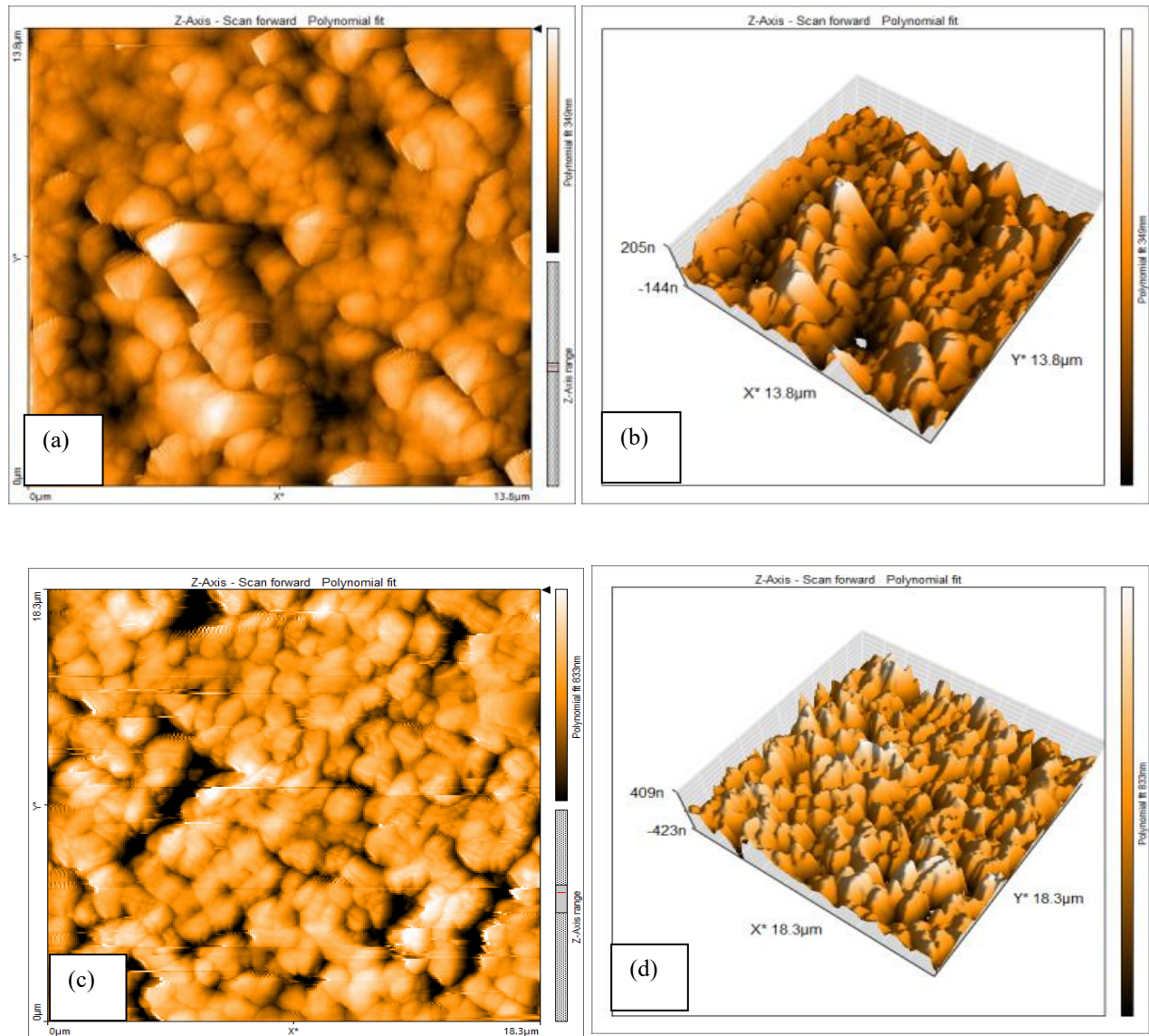


Figure 6 2D and 3D images of (a) and (b) for PS; (c) and (d) for PS after adding G NPs using drop casting method respectively.

4.6. UV-vis

The absorption spectrum is very useful to provide accurate information on film material that is necessary for the study or design of devices. The optical properties of materials depend on their type, crystal structure as well as surface roughness, thickness and surface defects responsible for scattering the incident beam. UV-vis spectra of graphene with different concentration (solution concentration 60%) in the range of (210-1100) nm have been examined as shown in Figure 7 [7]. More concentrated solutions contain a greater number of molecules that interact with the light that penetrates, thereby enhancing their absorbance. Since the film cannot be utilized as a window layer in some applications at the UV wavelength, the transmittance falls in this region [13,14].

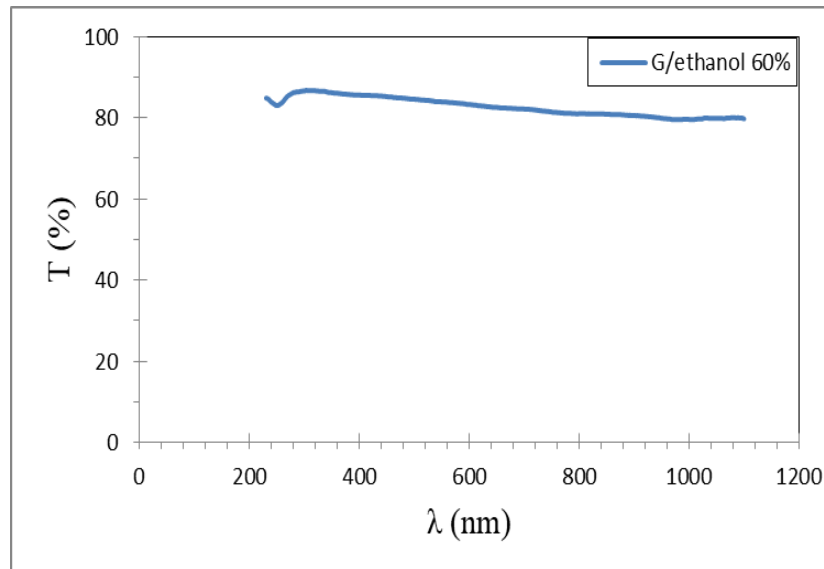


Figure 7 UV-vis spectra of G NPs with ethanol 60 ml.

The reflectance spectrum of the films prepared at a concentration of 60% is calculated by adopting the energy conservation law. Figure 8 shows the spectrum of the reflectivity of graphene prepared at different concentrations by thermal decomposition method in range (210-1100) nm. It notices that the reflectivity decreases in the case of concentration 60% this indicates that the surface roughness is increase with increasing concentration thus increase of Absorbance. The reflectivity of the samples is recorded to 0.08 at 210 nm for concentration 60%. The behavior of the refractive index varies depending on the preparation conditions and the technology used to prepare the substance. Its value depends on several factors, such as the type of material and crystalline structure. The refractive index (n) spectra of graphene with varying concentration within the range of wavelengths (210- 1100) nm have been shown in Figure 9. The nature of the refractive index curves is almost similar to the reflection spectrum curves the nature of the correlation between them according to the relationship. It is observed from the figure that the refractive index of concentration 60% it is 1.1 at 210 nm due to the increase in absorbance at the expense of transmittance and reflectivity [5].

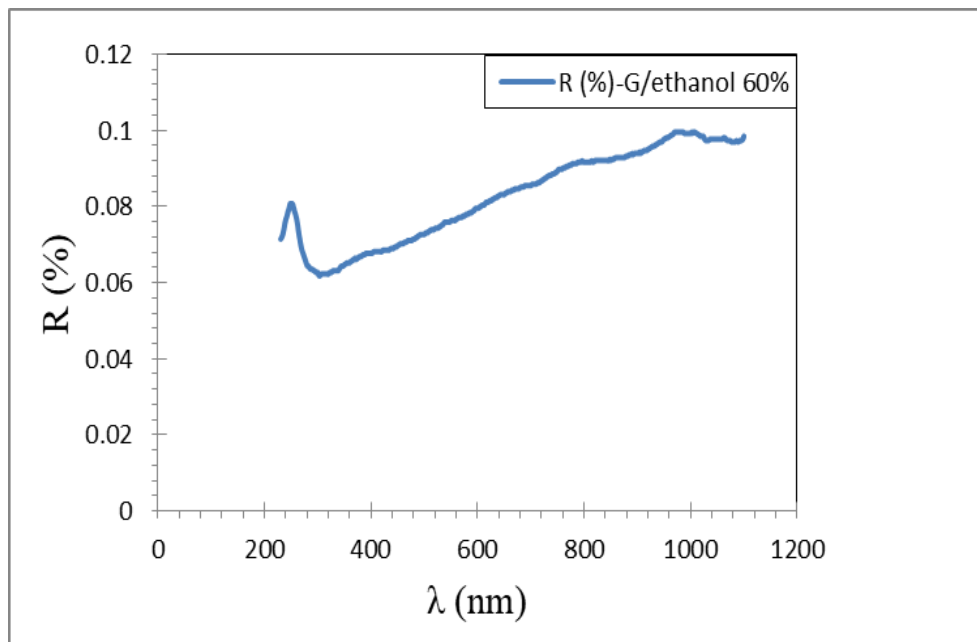


Figure 8 Reflectance spectra of GNPs with different wavelength λ at 60%.

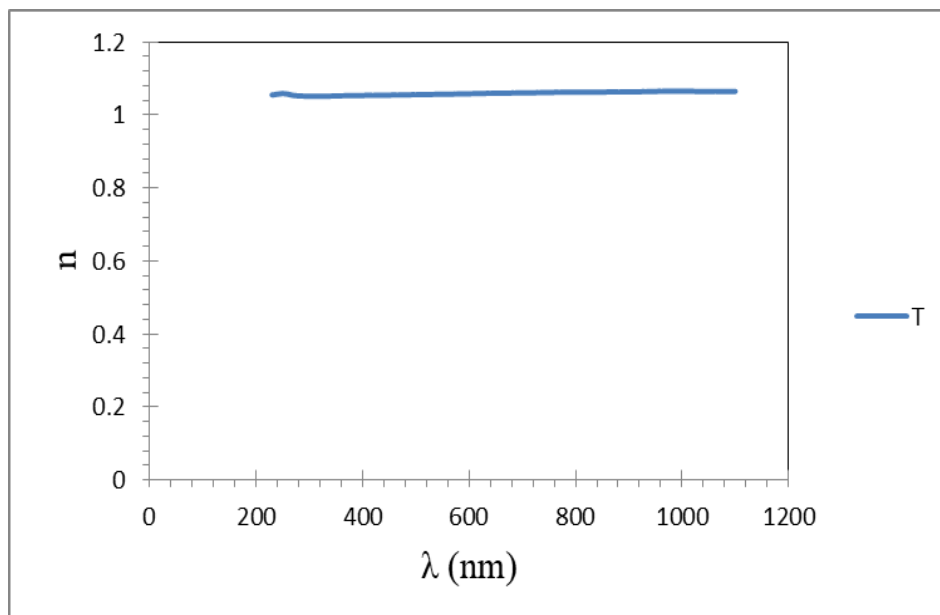


Figure 9 Refractive index of GNPs with different wavelength λ at 60%.

4.7. Electrical properties

Figure 10 depicts the electrical properties I-V analyses of the PS and GNPs:PS J-V. The value of the current has increased with the increase of voltage values, and the highest value is for the GNPs:PS, due to the high conductivity described by the GNPs:PS, which has led to an increase in the current relative to the samples of the PS and below the Silicon bulk, which has a high crystalline structure. It has been observed that the results of the current-voltage characteristics indicate that the value of the electric

current has increased for samples prepared using the drop casting method, resulting in an increase in the energy gap due to a reduction in sample size [8].

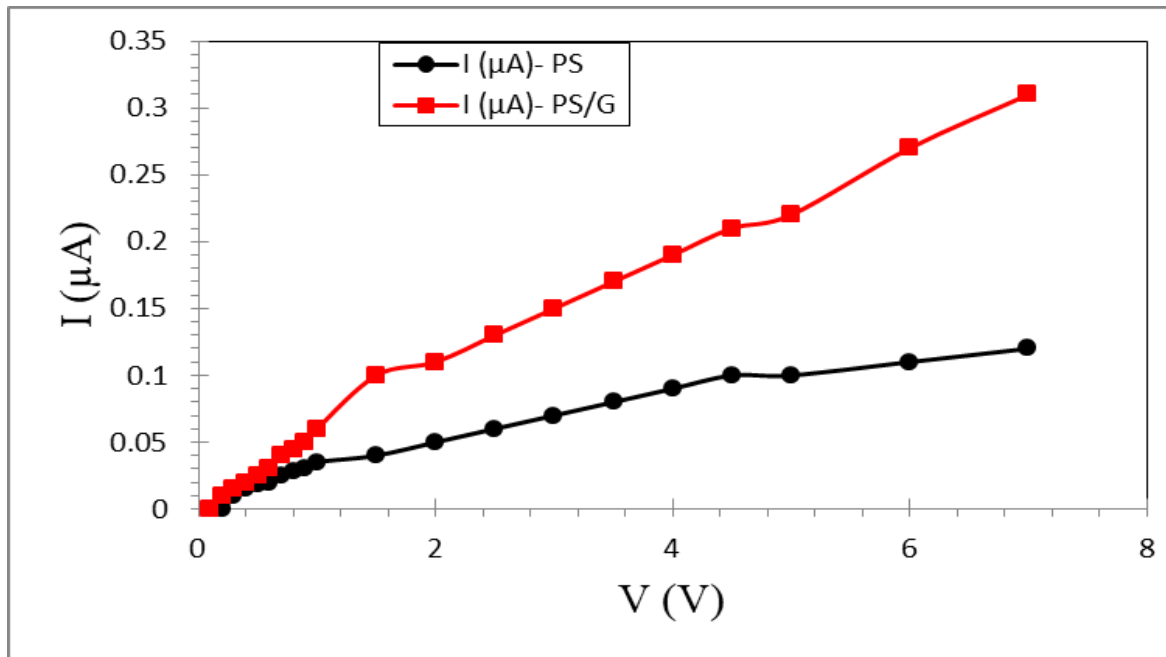


Figure 10 I–V properties of the preparation of the (A) PS and (B) GNPs:PS at various supply voltage.

4.8. Thermal properties

Figure 11 A and B shows thermal properties of the PS and GNPs:PS at different voltage values value from (0.2 and 0.6) V; its noticed that the current has been increased with the increase of voltage values and the highest current values is for GNPs:PS layer corresponding to PS. Figures 12 A and B show the thermal characteristics of porous Si (PS) and graphene (G).In the observed voltage range of 0.2 to 0.6 V, it is seen that the current exhibited a positive correlation with increasing voltage values. Notably, the greatest current values are recorded for GNPs.The PS layer that corresponds to the PS layer. Figure 12shows a system setup that used for the thermal properties.

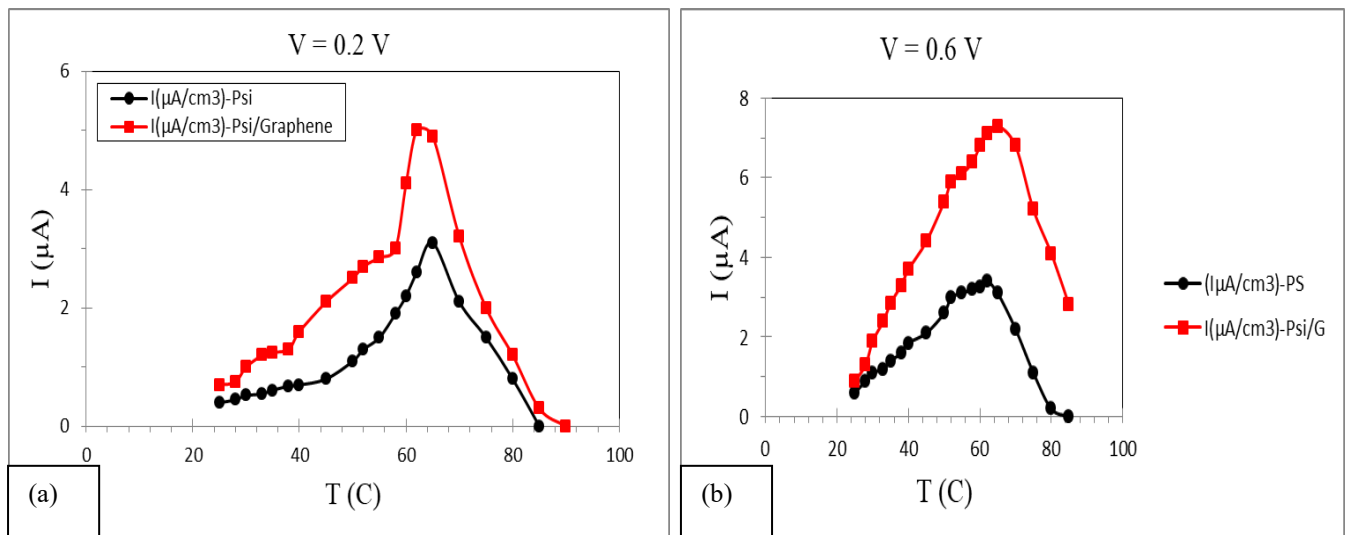


Figure 11 The thermal properties of (a) PS and GNPs:PS at 0.2 Volt, (b) PS and GNPs:PS at 0.6 Volt.



Figure 12 Thermal properties setup for PS and G: PS at various supply voltage after using welding method.

In the particular case of graphene deposition on porous silicon through drop-casting, the observed phenomenon, characterized by an initial peak in the current curve followed by another drop to zero as temperature rises, can be revealed by taking into account the individual characteristics of graphene and porous silicon, as well as their relationship with temperature. The topics of interest include thermal activation and mobility in graphene, thermal expansion and strain, defect generation and carrier scattering, and thermal degradation and phase transitions or melting. It is essential to acknowledge that the specific behavior will be contingent upon a variety of aspects, including the quality of the graphene, the attributes of the porous silicon, the relationship between the two substances, and the testing parameters. In order to get a thorough comprehension and elucidation of the observed phenomena, it is very probable that a complete investigation would need the integration of experimental measurements, materials characterization, and theoretical modeling.

5. CONCLUSION

Photo electrochemical etching (PECE) was used to make porous silicon (PS) on an n-type silicon (Si) wafer at a current density of 15 mA/cm^2 for 15 minutes. The XRD Examination show, all the samples presented a well-defined crystalline phase of PS and GNPs with a (211 and 002) peak respectively as a preferred orientation. While the grain size of the films is increasing with the decreased in GNPs from 72.5 to 43.5 nm. Additionally, the SEM morphology images demonstrate that the GNPs have a significant impact on the surface morphology. Furthermore, UV-vis spectroscopy results indicated that the optical transmittance spectrum decreases in UV region. FTIR transmittance spectra revealed the presence of peaks in GNPs are caused by oxygenated functional group that were produced when grapheme was successfully oxidize. The electrical measurements revealed that the electric current for samples prepared using the drop casting method has increased with the voltage increased. Finally, for thermal properties, the current increase with the increased of temperature at maximum point then decreased to zero with the voltage increased for both voltage values 0.2 and 0.6 V.

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