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RESEARCH ARTICLE - Physics

Structural and optical properties of black silicon prepared by wet chemical etching

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Abstract

The study focuses on the optimization of black silicon (B-Si) fabrication via etching using a solution composed of KOH, NaOH, and Na_2SiO_3 at constant concentrations and varying durations. Surface morphology was analyzed using Field Emission Scanning Electron Microscopy (FE-SEM). The results showed that pyramidal structures were formed on the silicon surface with heights between 1.2 and 4.1 µm. The uniform distribution was confirmed by AFM images. The surface roughness decreased as the etching time increased; it was measured at 14.03 nm for 15 minutes and 5.59 nm for 30 minutes. The properties of photoluminescence (PL) were evaluated using a spectrometer and it was found that peak emission shifts to lower energies (red shift) when etching time is increased. Optical reflectivity decreases sharply when etching time is increased. Additionally, the longer the etching duration, the greater the energy gap of the material which increased from 1.25 eV to approximately 1.4 eV.

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Keywords: Wet Etching; Black silicon; Photoluminescence properties; Surface morphology; energy gap.

Introduction

Silicon is gaining popularity due to its compact size, broad applications, ease of integration, and long life, but its high reflectivity and broadband gap pose drawbacks [1].

In contrast to polished silicon, black silicon material exhibits ultralow light reflection in visible and near-infrared bands due to its nanostructure and impurity doping, particularly in the near-infrared region [2]. Black silicon (B-Si) is a promising surface texture for optoelectronics due to its large acceptance angle and low reflectance over a broad wavelength range. Where, the incorporation of anti-reflection surfaces into current equipment can significantly enhance light collection efficiency, which will significantly impact optoelectronic devices in the future [3] . The modification of black silicon, or textured surfaces, was discovered in the 1960s and 1980s as an unwanted side effect of chemical etching and reactive ion etching [4]. In 1996, Harvard University physics professor Eric Mazur and his graduate students discovered "black silicon" by irradiating a silicon wafer with ultra-short pulses from a femtosecond laser, revealing a vast array of nanoscale spikes. Research on the morphology and structure of black silicon is extensive due to its significant applications in various fields. ***Xuewen Geng et al***, reported that the final surfaces display morphologies that vary based on processing parameters, including the presence of pyramidal textures and the duration of etch exposure [5]. ***Yuanjie Su et al***, achieved a needle-like micro-column structure of Si during the KOH etching step [6] . David Schmelz et al. describe that black silicon morphology is defined by its structure height and lateral correlation length [7].

On ordinary silicon surfaces, black silicon is created by chemically depositing nano- or microstructures [7]. Because black silicon has a distinct morphology that efficiently inhibits optical reflection while enhancing light absorption and dispersion, silicon foils seem black rather than the more common silver-gray color [8]. Because of its surface structure, black silicon has unique optical and electrical properties. Its tightly packed micro-pyramids or nano-spikes result in strong light absorption and low reflection rates.

The percentage of both reflection and absorption can be used to measure the effectiveness of Super Black Silicon. According to Prabhat K. Agnihotri et al [9] reflection should be less than 2% and absorption should be greater than 99%.

Due to these characteristics, black silicon is perfect for a wide range of uses, such as Li-ion batteries and photovoltaics [9], [10], [11]. Black silicon's high surface area contributes to its conductive qualities by lowering recombination rates, which prolongs the life of charging stands[2]. Black silicon material is highly useful for optoelectronic devices because it exhibits photoluminescence properties and has high absorption coefficients in the visible and near-infrared ranges. Its high surface roughness results in low surface reflection, but it also increases light trapping, which boosts efficiency[12], [13], [14] . Black silicon, created by laser treatment (nano,femto-second laser) of crystalline silicon, reveals two bands of luminescence: red and green [9] .According to research by Md. Yasir Arafat et al., conversion efficiency can rise by as much as 22.04% when pyramidal structures' etching angle is increased to about ~70 [15]

A material such as black silicon is suitable for use in photonics and optoelectronics for the reason that it has distinct electrical and optical properties. The production methods of nanostructured black silicon plasma etching [16] reactive ion etching [17]metal assisted chemical etching [18]laser irradiation [19], and wet etching [20]. Among these techniques, wet chemical etching stands out as a low-cost and highly effective method. Unlike other approaches such as MACE which require expensive noble elements like Ag, Au, Pd, or Pt for production at mass scale— wet chemical etching does not involve costly materials [21].

Research work on the process of developing black silicon has been documented by various authors including Hsu in 2020 [22]; an example is the current study that fabricates nanostructured pyramidal black silicon using metal-assisted chemical etching with $AgNO_3$ as a catalyst. The study investigates how the concentration of $AgNO_3$ affects the passivation process and optical properties of black silicon. From the experiments, it is revealed that with an $AgNO_3$ concentration of 0.03 M, the nanostructure length is around 300 nm. Moreover, the reflectance of black silicon with SiN_x and Al_2O_3 stack is 0.8%, showing similar results to conventional black silicon with micrometer-sized nanowires. In 2021 Arafat, M. Y. [15]This paper introduces a novel, straightforward, and low-cost approach to texture monocrystalline Si (100) wafers for creating a micro-pyramidal structured layer. The wet chemical anisotropic etching technique using isopropyl alcohol and potassium hydroxide solution generates elongated micro-pyramidal structures on the surface— leading to optical confinement effect by textured pyramids having heights of 1–2 μm and 70-degree angles that significantly reduce B-Si wafer reflectance.

Thus, this study aims to produce black silicon by using low concentration wet chemical etching solutions including potassium hydroxide (KOH), sodium hydroxide (NaOH), and sodium silicate (Na_2SiO_3). The objective is to get black silicon with a pyramidal structure. Various analytical methods have been used to study and describe the key characteristics of the black silicon generated, such as its surface morphology, surface topography, and structural and optical properties. This work presents a novel method for the preparation of black silicon with promising structural and optical properties for Optoelectronic applications such as solar cells and photodetectors without an auxiliary catalyst such as (Ag, Au, Al, etc.), unlike previous methods that used auxiliary metals which are considered expensive.

Experimental

Alpha Chemika supplied ethanol (C_2H_6O; molecular weight: 46.07 g/mol; purity: 99.99%) and isopropanol (C_3H_8O; molecular weight: 60.10 g/mol). HIMEDIA supplied hydrofluoric acid (HF) with a molecular weight of 20 g/mol and a concentration of 40%. EDUCEK Chemicals India supplied us with potassium hydroxide (KOH) with a molecular weight of 56.1056 g/mol. The sodium meta silicate

(Na2SiO3.9H2O; molecular weight: 284.2 g/mol) was ordered from Central Drug House (P) Ltd. Sodium hydroxide (NaOH; molecular weight: 40 g/mol) was ordered from ACS Chemicals.

The wet chemical etching procedure was used to generate black silicon on n-type <100> wafers with dimensions of (0.5×0.5) cm^2, a resistivity of ρ~ 1-10 Ω.cm, and a thickness of 500 μm. Initially, silicon wafers were ultrasonically cleaned for 10 minutes at room temperature using an alcoholic solution (40 percent isopropanol and 60 percent acetone). To get rid of the undesirable native oxide layer, the wafers were submerged in a solution made of HF and water at a 2:10 ratio. To counteract the effects of the alkaline solvent ($KOH+NaOH+Na_2SiO_3$), the etching solution is made from KOH, NaOH, and Na_2SiO_3 (3 Wt.%, 3 Wt.%, and, 1.5 Wt.%) respectively at various periods (15 and 30 minutes) and at a temperature of 80 ℃. The wafer is cleaned with DI water after etching and drying by a hot air stream.

In order to stop oxidation, the samples are lastly kept in ethanol. The hot plate stirrer device is used to regulate the temperature of the etching solution. A Field Emission Scanning Electron Microscope (FE-SEM) (Inspect f 50-FEI business) was used to evaluate the surface morphology. An Atomic Force Microscope (AFM) model (TT-2) was used to assess the surface roughness, maximum height, and roughness average. The X-ray diffraction of the black silicon nanostructure was measured using AERIS-XRD, a PANalytical business of Cu-Kα radiation at λ = 0.15406 nm. Utilizing a 150W continuous wave xenon-arc lamp, the Fluoromate FS-2 spectrometer was used to analyze the photoluminescence (PL) characteristics of black silicon. The preconfigured Raman spectrometer equipment at 532 nm was used to test the Raman characteristics of black silicon. A UV-VIS.NIR (Jasco V670/Japan) was used to examine the black silicon reflective spectrum

Results and discussion

The morphology of the black silicon surface was studied using FE-SEM (top view and cross section) images at two magnifications of 5μm and 10μm as shown in Figs. 1a and b. A cross-section FE-SEM images of Figs.1a and b show that the morphology of pyramids of varying sizes is dispersed throughout the entire surface of the etched silicon, as well as, a uniform distribution of pyramids with heights ranging from 1.2 to 4.1 μm, depending on the etching time. And that agree with [5]. The etch rate was determined to be 0.168 and 0.088 μm/min at 15 and 30 minutes of etching durations, respectively, and that explains the different shapes and heights of pyramids. This behavior is in agreement with [7].

Micro-pyramidal structure formed by etching silicon substrates with an etching solution produces a black silicon layer that absorbs light by more than 95% due to the long interaction between light and the pyramids, which causes light to be scattered in a random manner as it reflects multiple times between the pyramids. The surface reflections of the incident rays led to an increase in light absorption[15].

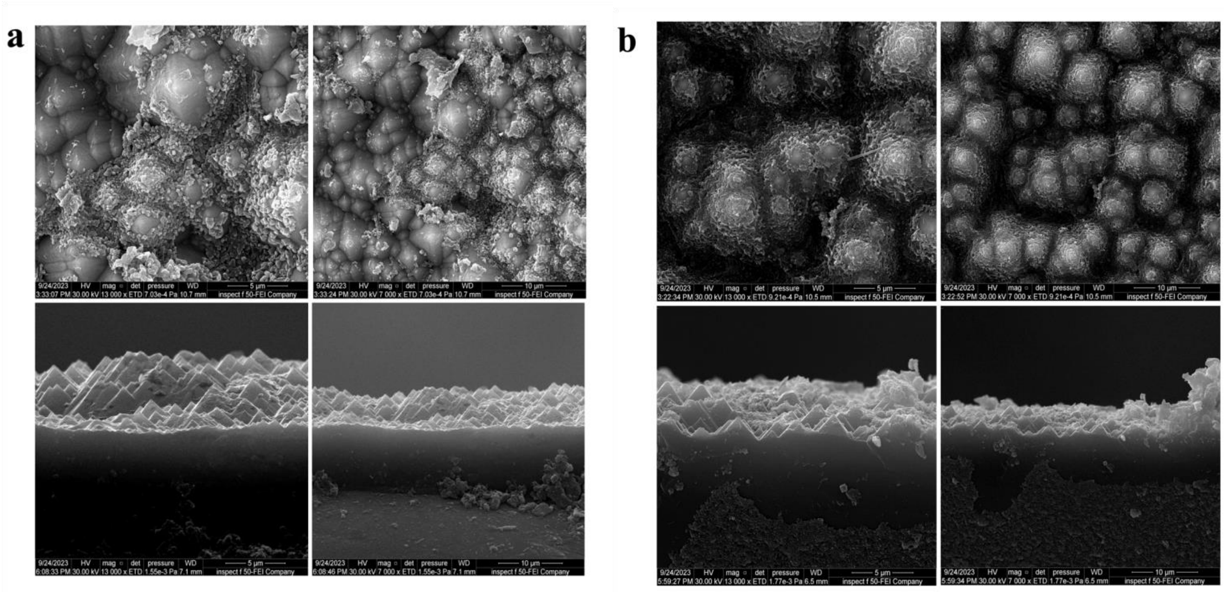

Fig. 1: FE-SEM images top view and cross section of black silicon Prepared at different etching time (a) 15 min, and (b) 30 min.

Figures 2a and 2b show AFM pictures of black silicon (B-Si) samples with different etching periods. These photos show pyramidal structures and their uniform distribution throughout the surface in 3D and 2D topographies. The pyramid matrix growth shows that etching can shape silicon into nanostructures. The consistent and periodic distribution of these pyramids shows manufacturing repeatability and control. Quantitative surface roughness research shows how etching time affects black silicon morphology. Surface roughness was 14.03 nm and 5.59 nm for 15- and 30-minute etchings, respectively. This result is agreement with [23].

The silicon surface is smoothed by longer etching time, reducing surface roughness. The roughness numbers also indicate that the black silicon surface has strong texturing, which increases surface area and scattering to improve light absorption. Table 1 provides average roughness (S_a), RMS surface roughness, and maximum height (S_Z) of black silicon samples generated under varied etching settings to support these results. This tabular data compares surface morphological factors to help explain how etching parameters affect surface characteristics. [24]

Table 1: Roughness parameters of black silicon at different etching time.

Sample	RMS (nm)	Sz (nm)	Sa (nm)
15 min	14.03	154.92	9.80
30 min	5.59	111.48	2.90

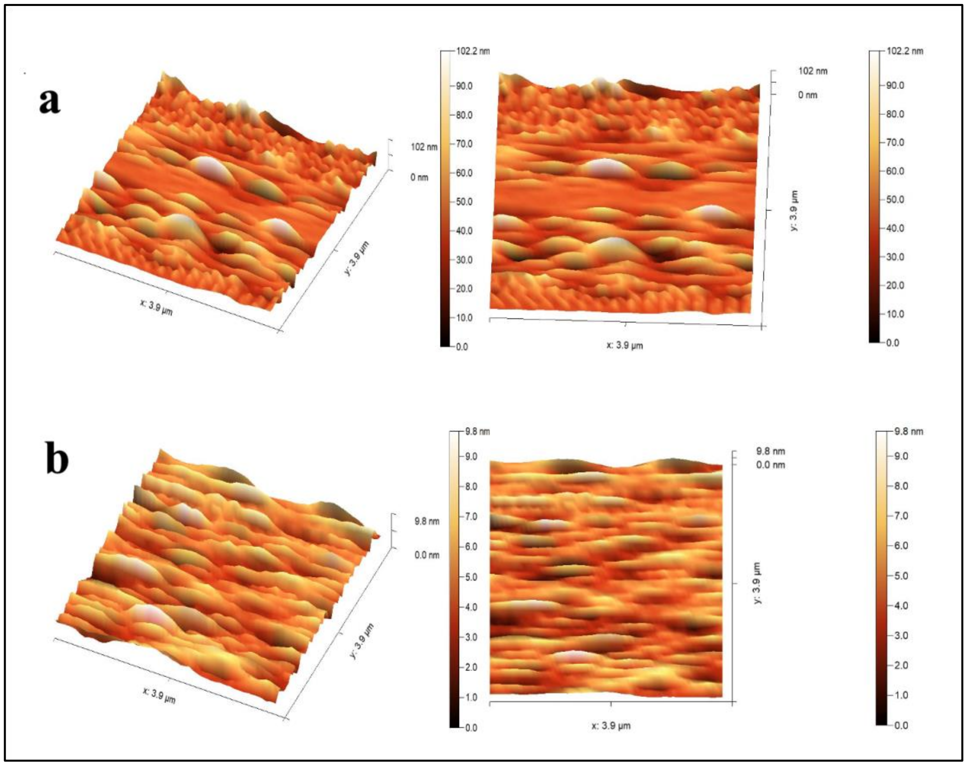

Fig.2: 3D and 2D AFM images of black silicon prepared at different etching time (a) 15 min, and (b) 30 min.

The X-ray diffraction patterns of black silicon that were created at various etching durations are shown in Figure 3. The study's findings demonstrate the crystalline structure of the samples that were etched at 15, and 30 minutes. Over the course of fifteen minutes, the pattern of the sample that was generated demonstrates a rise in the strength of the peak that is situated at 2θ = 69.3237º, which corresponds to the (400) plane. While the sample that was produced at thirty minutes demonstrates a lack of crystallinity, as shown by a peak that is situated at two-fold angle equal to 69.3045 degrees along the (400) plane. The determination of the crystal size of black silicon is accomplished by employing Sherer's equation [25].

$$L = \frac{K\lambda}{B\ COS\theta} \tag{1}$$

Where λ is the x-ray wavelength, k is the Sherer's constant of 0.9, B is half the peak width, and θ is the peak angle. The increased etching time leads to an increase in the size of the crystal. Due to the growth of more micro-pyramids, this leads to the formation of larger crystallites. At 15 minutes, the crystal size of black silicon was measured to be 63.41 nm; at 30 minutes, the crystal size increased to 83.46 nm [26].

The development of nanostructure morphology is responsible for the marginal change in lattice constant that happens as etching time rises. The size and dispersion of the nanostructure crystallites control the strain that the mechanical forces this procedure places on the silicon cause. Table 2 shows the black silicon crystallite size, FWHM, lattice constant, and strain as a function of etching time.

Table 2: Black silicon XRD analysis results for various etching times.

Etching time (min)	2θ (Deg.)	FWHM (Deg.)	d_{hkl} (Å)	L (nm)	hkl	$a_{B\text{-}Si}$ (Å)	strain
15	69.3237	0.1523	1.35442	63.41	400	5.4176	0.000428
30	69.3045	0.1157	1.35474	83.46	400	5.5189	0.000191

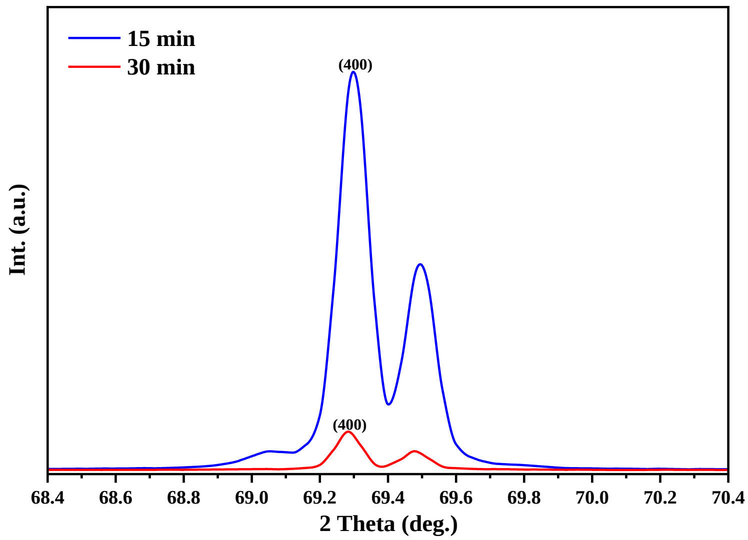

Fig. 3: Black Silicon XRD patterns generated at various etching times

Figure 4 displays the photoluminescence spectra of black silicon fabricated by the wet etching process. The photoluminescence (PL) spectra of the sample, which was developed for a duration of 15 minutes, exhibit a solitary wide-ranging band that is centered at 677.6 nm. This band corresponds to an energy of about 1.83 eV and has a full width half maximum (FWHM) of 240.25 nm. The sample produced for 30 minutes has a peak at a wavelength of 720.9 nm, which corresponds to an energy of about 1.71 eV and a full width at half maximum (FWHM) of 208.09 nm. The relationship between etching time and the peak of photoluminescence is evident. The peak shifts toward longer wavelengths as the etching time increases; this is referred to as redshift. This shift is attributed to the larger size of crystals present on the surface of black silicon. Importantly, these findings align with the results obtained from X-ray diffraction (XRD) analysis [27]. Furthermore, an extended duration of etching results in the creation of many micro-pyramids, which are helpful in capturing light. Consequently, quantum confinement arises due to enhanced light absorption. Table 3 presents the recorded PL Peak Emission, Energy gap, and FWHM of the peaks based on the etching time. And the precision of PL is used to estimate the band structures of nanostructures, calculating the energy gap based on the emission spectrum. While this accuracy is lost when calculating the energy gap based on reflectivity because of the nature of the surface and the various absorption and impurity regions. Where, a band gap value is typically determined by comparing the light-onset energy of reflectance (R) to the lowest energy in photoluminescence (PL) [28].

Table 3: Analysis of photoluminescence (PL) data pertaining to black silicon (B-Si).

Etching time (min)	Peak Emission (nm)	Energy gap (eV)	FWHM (nm)
15	677.6	1.83	240.25
30	720.9	1.72	208.09

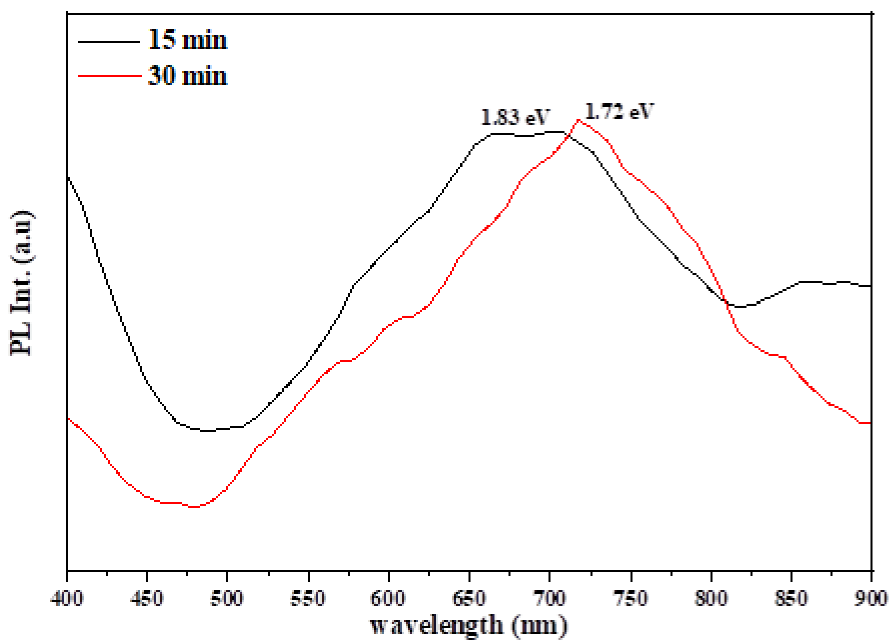

Fig. 4: The impact of etching time on the photoluminescence spectrum of wet chemically etched black silicon.

Figure 5 shows the Raman spectra of black silicon samples etched at different times, revealing the material's structural and vibrational features. The spectras unique peaks reveal black silicon's crystalline structure and chemical makeup. After 15 minutes of etching, the Raman spectra shows three distinct peaks at 520, 762, and 1032 cm^{-1}. These peaks represent structural vibrational modes. In diamond cubic lattice structures, the peak around 520 cm^{-1} indicates that silicon retains its crystalline form after etching. The peak at 762 cm^{-1} is the first-order optical phonon mode (TO) of silicon, indicating Si-Si bond stretching vibrations in the crystal lattice. The peak at 1032 cm^{-1}, corresponding to the second-order optical phonon mode (2TO) of silicon, suggests flaws or disorder in the crystal lattice, presumably caused by etching. The Raman spectra of the material etched for 30 minutes shows distinctive peaks at 520, 762, and 1032 cm^{-1}, with possibly changed intensities or shifts. The stability of these peaks over multiple etching durations shows that the black silicon structure is stable and resistant to severe etching changes. These Raman peaks reveal black silicon's structural development under various etching settings. The retention of crystalline silicon peaks demonstrates its basic structure, which maintains its electrical and optical characteristics.

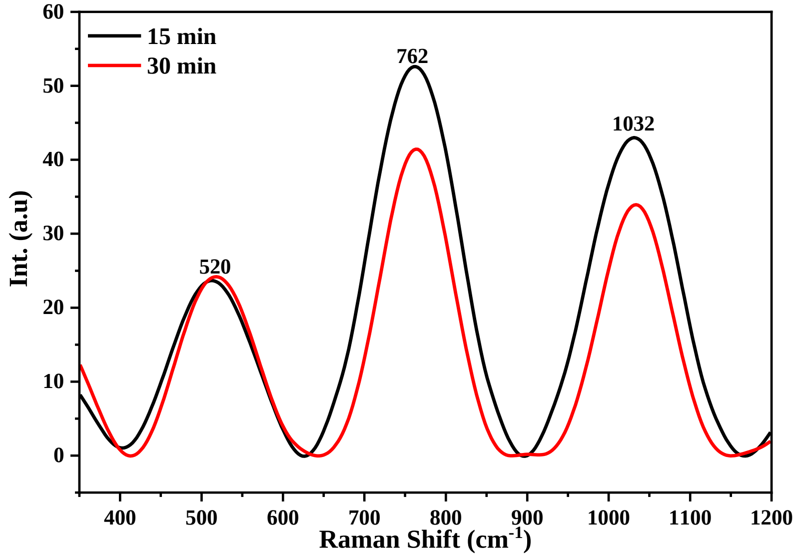

Fig.5: The Raman spectra of black silicon prepared at various etching times.

Figure 6 shows how etching time affects black silicon optical reflection. Data shows significant patterns throughout spectral bands, revealing the material's light absorption ability. First, the 15-minute-etched black silicon sample's reflection spectrum is noteworthy. The UV range has low reflectance, roughly 4%. This shows that surface morphology has already reduced reflection in this high-energy location after a short etching period. Reflection drops to 3% in the visible range. This decrease suggests that pyramidal structures formed during etching improve surface morphology. These structures disperse light, reducing reflection and improving light absorption. The 30-minute-etched black silicon sample improves optical performance. A 1.5% drop in reflection across all spectral areas relative to the 15-minute etched sample is seen. Extended etching durations improve surface morphology and light absorption, as seen by this decrease. Light scattering pyramidal structures on the surface become more prominent and efficient, decreasing reflection and increasing absorption. Black silicon absorbs incoming light effectively over a wide range, acting more like a black body. Low reflection is crucial in optoelectronic device applications. Photodetectors, solar cells, and optical sensors work better with black silicon because it absorbs light and minimizes reflection. For black silicon to be fully used in optoelectronic applications, etching parameters, particularly time, must be optimized. Enhancing etching procedures and understanding their mechanics will improve the field and provide new opportunities for black silicon-based technology.

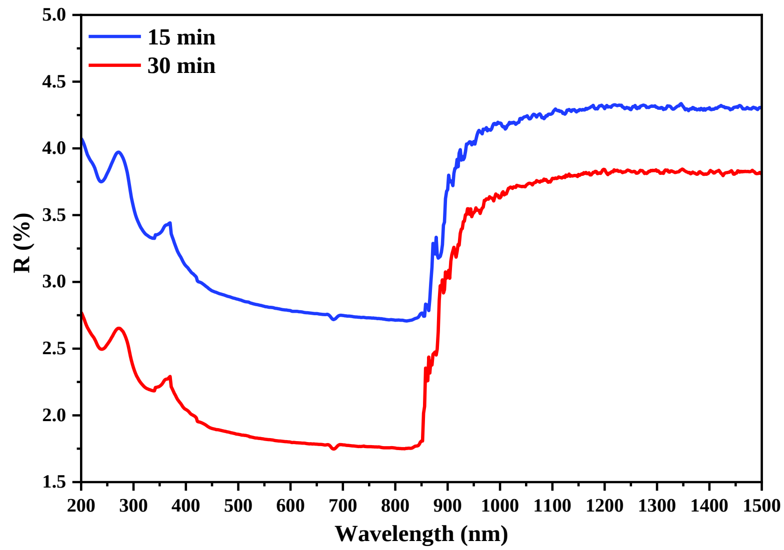

Fig. 6. Black silicon (B-Si) reflectance spectra generated at various etching periods.

The band gap for black silicon can be obtained from a reflection spectrum by the Tauc method Where the reflection spectrum is converted to the Kubelka-Munk function F(R∞) [29].

$$F(R_\infty) = \frac{1}{2R_\infty}(1 - R_\infty)^2 \qquad (2)$$

the energy gaps of the samples were obtained. The energy gap of the sample prepared at a time of 15 minutes was 1.25 eV, while the sample prepared at a time of 30 minutes had an energy gap of around 1.4 eV. This suggests that the band gap of black silicon is increasing with increasing etching time, as illustrated in Figure 7.

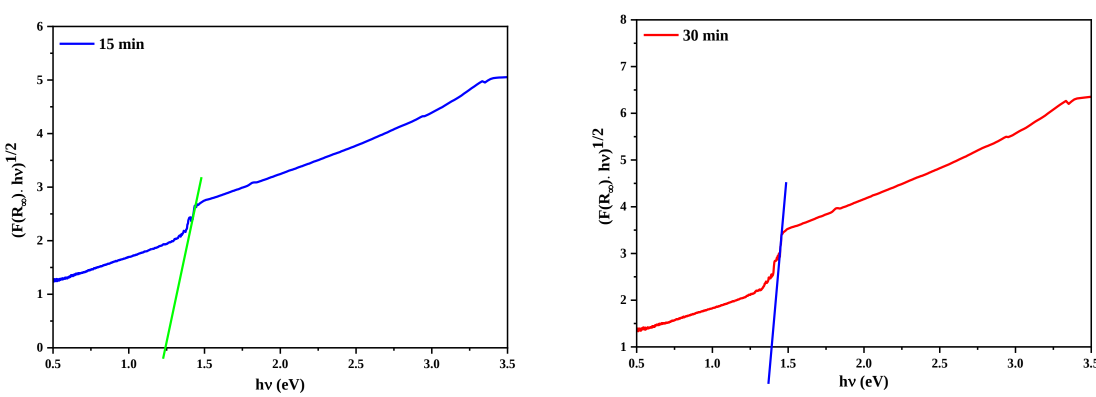

Fig.7. Energy gap of black silicon prepared at different etching times (a) 15 min and (b) 30 min.

Conclusion

This study successfully fabricated black silicon using the chemical etching technique. The effect of etching time on the morphological, structural, and optical properties of nanocrystalline black silicon was examine using various techniques, including FE-SEM, AFM, XRD, and PL spectroscopy. Chemical Etching with a pure mixture of KOH, NaOH, and Na_2SiO_3, without any metal-assisted etching, produced nanostructures with pyramid matrix growth and high absorption that are better suited for use in optoelectronic applications. As well, the etching time can be adjusted to change the energy gap, suggesting potential applications in photovoltaic and sensing technologies. Where, the surface nanostructure of black silicon is the primary factor influencing its optical properties, as determined by optical characterization.

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