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Experimental Analysis of Anisotropic Surface Texturing Process of Crystalline Silicon Wafers

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ABSTRACT

The development of silicon devices, circuits, and systems in most cases relies on the wet-chemical etching of silicon wafers (SWs). To achieve deep etching and micromachining, shaping, as well as cleaning, the dissolution of silicon using liquid chemical solutions is imperative. This study reports an experimental investigation of surface texturing of silicon wafers using a mixture of aqueous potassium hydroxide (KOH) solution and isopropyl alcohol (IPA) as a complexing agent to enhance light absorption and reduce the optical reflectance in the visible spectrum. Crochralski (CZ) silicon wafers of 100 mm diameter, 2” <100>-oriented, n-type, resistivity (Ωcm) of 7-21, with polished and lapped surfaces were utilized in the experiment. The process variables investigated included temperature (60 – 90) °C, duration of etching time (30 – 60) mins and concentration of KOH and IPA of (1 - 4) mg/l for KOH and (2 – 8) mg/l for IPA. The properties of the etched and unetched silicon wafers in terms of morphology, structure, photoluminescence, and electroluminescence were investigated to determine the effects of the process parameters on the efficiency and structural properties of the textured wafers. The SEM measurements revealed the presence of localized roughening pyramidal images. This showed that the use of KOH and IPA solutions on the silicon wafers revealed pyramidal structures that can be used to control the optical reflectance of the silicon wafers due to light scattering by the localized roughening. The applied etching procedure also produced low-reflecting materials whose reflectivity increases with wavelength. This study shows that textured material has great potential in optoelectronic device manufacturing processes.

1. Introduction

The study of crystalline silicon has a very important role in modern science and technology due to its excellent properties. It is generally known that about 90% of the solar panels used worldwide are comprised of silicon-based cells [1]. The high refractive index exhibited by silicon usually leads to optical losses in solar panel manufacturing. Mostly, the rays absorbed from the sun are moderately weak [2, 3]. Due to these optical losses, the efficiency of crystalline silicon solar cells reduces to a level which is usually between 12 and 20 % [4]. There have been concerted efforts geared towards improving the efficiency of solar cells based on silicon materials by exploring greater opportunities in developing reflection-reduction technologies to enhance better absorption of incident sun rays and invariably, improve crystalline solar cell [4 -7]. Though the thermodynamics of these solar cells are not easy to figure out, they have been extensively utilized in the production of optoelectronic devices [8]. There are also frequent and increasing applications in nano-electronics, communications, solar cells, etc [5, 9]. However, of all the anticipated applications of crystalline silicon, solar cells (photovoltaic) are the most attractive form owing to their advantages over conventional sources of energy (fossil fuels) [9]. As an eco-friendly and clean supply of energy, the effects of pollution resulting from the incomplete combustion of fossil fuels are eliminated [10]. Unfortunately, the high optical losses and low efficiencies of silicon solar cells make them less competitive [9]. In achieving efficiency in silicon solar cell manufacturing, the requirement for the uniformity of textured surfaces is imperative. This can be undertaken through texturing the surface of the crystalline wafer using anisotropic etching to transform or alter the silicon's surface parameters into different structures. In so doing, there is a reduction in reflection and enhancement in the absorbed incident light onto the silicon wafer [5, 11].

In several studies [12 -14], it was indicated that the nature and amount of IPA and the complexing or wetting agent (KOH) used determines the uniformities and sizes of structures (pyramidal) formed. Additional studies revealed that the IPA concentration had a strong effect on the surface roughness of <100> -single crystals at 80 ℃ with optimal KOH: IPA concentration of 2:4 [11, 12]. However, the influence of surface texturing on the light-trapping properties of silicon wafers has been severally investigated [13, 15 – 17]. Results showed optimum parameters for temperature, etchant concentration and time with reasonable values for surface roughness, and comparative optical reflectance in the visible spectrum of less than 10%. In their study, Fashina et al [5] investigated the surface textured of silicon substrates. The inherent study showed a surface with an average roughness of 593 nm using the optically textured silicon wafer. This result was in agreement with other studies conducted by Dobrzanski and Drygala [8] and Salwa et al [18]. Furthermore, analytical models were developed to investigate the optical reflection behaviour of alkali-textured SWs under a non-normal incidence angle [19, 20]. Consequently, it was observed the reflectance angles were reduced more at lower angles of incidence, when compared to those on flat substrates. The result was in accordance with other earlier studies in literature [13, 18].

Mechanical and chemical processes are often used in the preparation of polished silicon wafers where the silicon single-crystal ingots are sliced into circular disks referred to as wafers and then flattened using the process of lapping which involves scrubbing the wafers with abrasive material [21]. Etching is used to remove any mechanical damage which occurs during the mechanical shaping processes. Most often, etching process precedes other unit operation processes such as polishing and cleaning before device construction activities. Chemical etching of SWs is often achieved by bathing the wafers in an etchant which could be an acidic solution of HNO_3 + HF and KOH [11, 15]. One of the several factors considered in the reduction of the conversion efficiency in crystalline silicon solar cells is optical losses which are mainly due to surface reflection. In mitigating this challenge, anti-reflection (AR) and surface techniques have been developed. Consequently, texturing process is undertaken to improve surface morphology for reflectance reduction. Most often, alkali hydroxide etchants such as KOH or NaOH have been used for surface texturing of crystalline SWs. These etchants are mixed with alcohol additives which produce anisotropic etching properties that cause orientation difference between the <100> and <111> planes. These etching mixtures give rise to changes in the etching rate, as a result, random pyramids are formed by the intersection of the <111> planes and aligned to <100>. The most popular solution used for texturing process is KOH mixed with isopropyl alcohol (IPA) [5, 11, 15]. In semiconductor industries, anisotropic etching using alkaline solutions at high temperatures is often employed to pattern single-crystal SWs. Characterization of SWs of different crystalline orientations and doping densities using etching processes have severally been investigated [11, 15, 22].

To enhance the efficiency of silicon solar cell (SC) technologies, surface texturing of the crystalline silicon system is necessary [9]. The application of alkaline solutions of either NaOH or KOH and IPA is highly preferred and common for anisotropic etching of crystalline-SWs [15]. Etching a textured surface of microscopic pyramids on SWs with <100> - oriented surfaces have been implemented with relatively mild alkaline solutions whose concentration is below 5% using either KOH or NaOH. In solar cell developments, the textured surface finds application in the minimization of reflection losses from the front surface to enhance optical light-trapping within the silicon cells that possess reflective back surfaces [12].

Surface texturing of silicon wafers using chemical processes has not been adequately characterized as much as those implemented for thinning in the semiconductor industries. Generally, alkali solutions, such as KOH or NaOH, have been often applied to the surface texturing of crystalline silicon systems [24]. Other alkali solutions, like LiOH, CsOH and NH4OH, have also been reportedly used similarly in literature [24]. To produce a different orientation between the <100> and <111> planes, the various alkali solutions are mixed with an appropriate chemical (alcohol) addition [5] Etching with such a combination brings about a modification in the etching parameters and, consequently, enhances the surface properties of the silicon system. The surface texturing of SWs could improve the absorbance characteristics which in turn, increase the photo-conversion efficiencies of the cells [5, 11, 15].

Recently, researchers in crystalline silicon have taken a keen interest in investigating the use of mixed alkali solutions and alcohol to improve efficiency, with the goal of better understanding and manufacturing crystalline silicon-based solar cell application devices [5, 8]. Park et al [24] studied the effects of tertiary-butyl alcohol (TBA) on crystalline silicon for solar cell applications. In their report, they indicated that a mixture of KOH and TBA was used to texture the silicon wafers. Also, Dubey and Gautam [9] employed a mixture of hydrofluoric acid, ethanol and deionised water in the ratio of 1:1:2. They found several improvements in the physical characterization of the treated silicon wafers. In all of these studies and several others, the role of IPA in the texturing process is not fully understood [12]. This has therefore necessitated the need for further studies to investigate the different combinations of these alkalines (IPA to KOH) which would safely be needed to influence the system, to reduce the reflection of incident rays on the silicon material while enhancing the light absorbance for photoelectric applications.

In the present study, to enhance and increase the efficiency of short circuit current devices, texturing of SWs were prepared by wet chemical alkaline etching with anisotropic characteristics using a mixture of KOH and IPA solutions. The effects of the textured process parameters (time, etchant concentration and temperature) on the surface structure and optical properties of the SWs were also determined and discussed.

2. Methodology

The procedures adopted for the texturing of the silicon wafers were those reported by Fashina et al [11] and King and Buck [6]. The texturing experiment was conducted using aqueous solutions of KOH and IPA as the complexing agents. The IPA facilitates the texturing process by dissolving hydrous silica formed at the reaction interface. Czochralski (CZ) silicon wafers of 1 μcm resistivity, polished and lapped surface were used for the investigation. The CZ silicon wafers used for the experiment were 100 mm in diameter, <100>-oriented, n-type doped, thickness of 250μm, resistivity (Ωcm) of 7-21, with the polished and lapped surface. The process variables investigated were temperature, time and IPA and KOH concentrations. The ranges considered were: time (30 – 60) mins; temperature (60 – 90C and etchant concentration (KOH – IPA): (1-4) mg/l – (2 – 8) mg/l. The study was conducted using a 250 ml flat-bottom flask containing the KOH solution. The etching process was performed at different etchant concentrations, times and temperatures in an electrical thermostatic water bath boiler with reciprocating motion.

The flask containing the KOH solution was heated on a hot plate equipped with a magnetic stirrer. The chemical solution included high-purity-deionized water (18MΩ.cm) which must be heated just to a temperature near the desired level before the addition of the complexing agents. The required concentration of the solution was prepared thus; The 100 g of KOH pellets were weighed into a beaker and IPA was introduced into the beaker, which was then made up into a 100 ml volumetric flask using high-purity deionized water (DI). This was prepared for a 40% concentration in the various volume ratios of KOH, IPA, and DI. Different concentrations of the various volume ratio solutions were prepared in situ accordingly. Thereafter, 40 ml of isopropyl alcohol (IPA) was added to the solution. The isopropyl alcohol increases the anisotropy in the etching process. Different concentrations and temperatures of the mixture solution were prepared accordingly.

Before the commencement of the texturing process, the wafers were first etched for 60 seconds in buffered-oxide-etchant (BOE) consisting of 0.5 % hydrogen fluoride (HF). Thereafter, surface-damage removal etches in a 1:1 solution of KOH: DIH2O at 85°C for 30 minutes were then used to thin the wafers to a dimension of 30 μm approximately. Furthermore, the wafers were then stored in an isopropyl alcohol solution for 10 minutes to prevent surface oxidation before the texturing solution attained the desired temperature. Thereafter, the wafers were then dipped in the texturing solution for the desired time duration, evacuated and further rinsed with deionized water and dried under a stream of nitrogen gas. Fresh solutions of deionized water, KOH and IPA were employed for each experimental study. After each experiment, the wafers were rinsed and dried in nitrogen before a number of performance measurements were carried out on each of the textured wafers.

2.1 Characterization

Wafer surface roughness and waviness were measured after etching using a surface profiler (Quad ProPlus Window, VeecoDektak Stylus Profiler). Spectral reflectance based on the photoluminescence of the textured-SWs was determined via an Avantes, AvaSoft 7.1 UV-Visible spectrophotometer while the scanning electron microscope (SEM) (EVO MA/10 MODEL) at different magnifications was used to determine the surface structure and morphology. To estimate the roughness and the reflectance of the resultant wafers, various points were measured and the average value was taken.

3. Results and Discussion

3.1 Surface Profilometry

Stylus surface profiler is a useful characterization technique for the determination of the size and surface roughness of Nano-materials. The technique aids in determining the presence of aggregates, voids, and homogeneity orientation with respect to the surface [4]. On the other hand, the roughness (surface) of any silicon wafer is an important parameter in semiconductor characterization. It influences the mobility as well as the optical and electrical properties of the wafer for device applications [11]. The average roughness of the etched wafers is given in Tables 1, 2 and 3 for the various properties investigated on the textured SWs. The values obtained at the various process parameters are in agreement with other studies conducted on the dependence of wafer reflectance and etching time [11, 15]. Table 1 indicates that the average roughness of the etched-SWs increased with the etching time. The gradual increase in surface roughness with etching times corresponds with the reflectance result of less than 10% achieved in literature for silicon materials [15].

Table 2 shows that there was a corresponding increase in the roughness of the etched-SWs as the etching temperature was increased. The magnitudes of the values are similar to those reported in the literature for etched silicon wafers [11, 15]

Table 3 shows the effects of varying the etchant concentrations in the etched silicon wafers. From table 3, it was observed that the average roughness of the wafers was significantly influenced as the etchant concentration varied around the optimum values of 2:4. The average roughness was affected by both the amount of KOH and IPA present in the reacting mixture as well as on the textured wafers.

From Tables 1 and 2, it was observed that the average roughness of the etched wafers was increasing correspondingly with time and temperature. The main effect of these parameters (time and temperature) on the surface roughness is significant, as higher time and temperature produced rougher surfaces. Similar observations have been reported in the literature and were due to the presence of defects within the wafers system [5, 20, 24]

Table 1: Etching time and average roughness of etched wafers

Time(Mins)	**Average Roughness,(nm)**
30	2008.71
40	5650.20
50	6211.85
60	6432.78

Table 2: Variation of temperature and average roughness of etched wafers

Temperature(^{0}C)	**Average Roughness (nm)**
60 ^{0}C	5019.80
70 ^{0}C	5743.65
80 ^{0}C	6413.71
90 ^{0}C	6606.45

Table 3: Varying etchant concentrations and average roughness of etched silicon wafers

Concentration (g/ml)	**Average Roughness (nm)**
KOH: IPA	
2 : 4	5650.20
1 : 2	5482.40
4 : 8	12865.51
1 : 4	6779.80
4 : 4	6558.71
2 : 2	8481.82
2 : 8	6819.67

3.2 SEM Analysis

Surface topology is used to understand the morphological characteristics or behaviour of etched silicon wafers and its role cannot be overemphasized. Scanning electron microscopy (SEM) morphology studies provide an understanding of the nature of the textured surface and are an important qualitative parameter in any study [5, 11]. Figure 1 shows the surface morphological patterns of the textured material at various etching times. Generally, it was observed that the materials were well-covered with different morphological structures. From the figure, the SEM textured images show an increased pyramid of grains that are randomly distributed. However, a further increase in textured time leads to the formation of various pyramidal grains with different morphological structures. Partially dense and compact nano-grains covering parts of the substrates are formed for A (Figure 1(a)). As the deposition time increases, the pyramidal grains increase in size with an agglomeration of clusters, as indicated in Figure 1(B) for B. After 50 minutes (Figures 1(C and D)), the clusters of larger grains metamorphose into smaller numbers of particle-like, rough, and irregular pyramidal grains. The development of various morphological features was due to the variation in nucleation rate as the texturing time increased [11]. This enhancement shows that varying the textured duration results in several pyramidal grains coalescing and diffusing simultaneously to form the variously defined morphologies

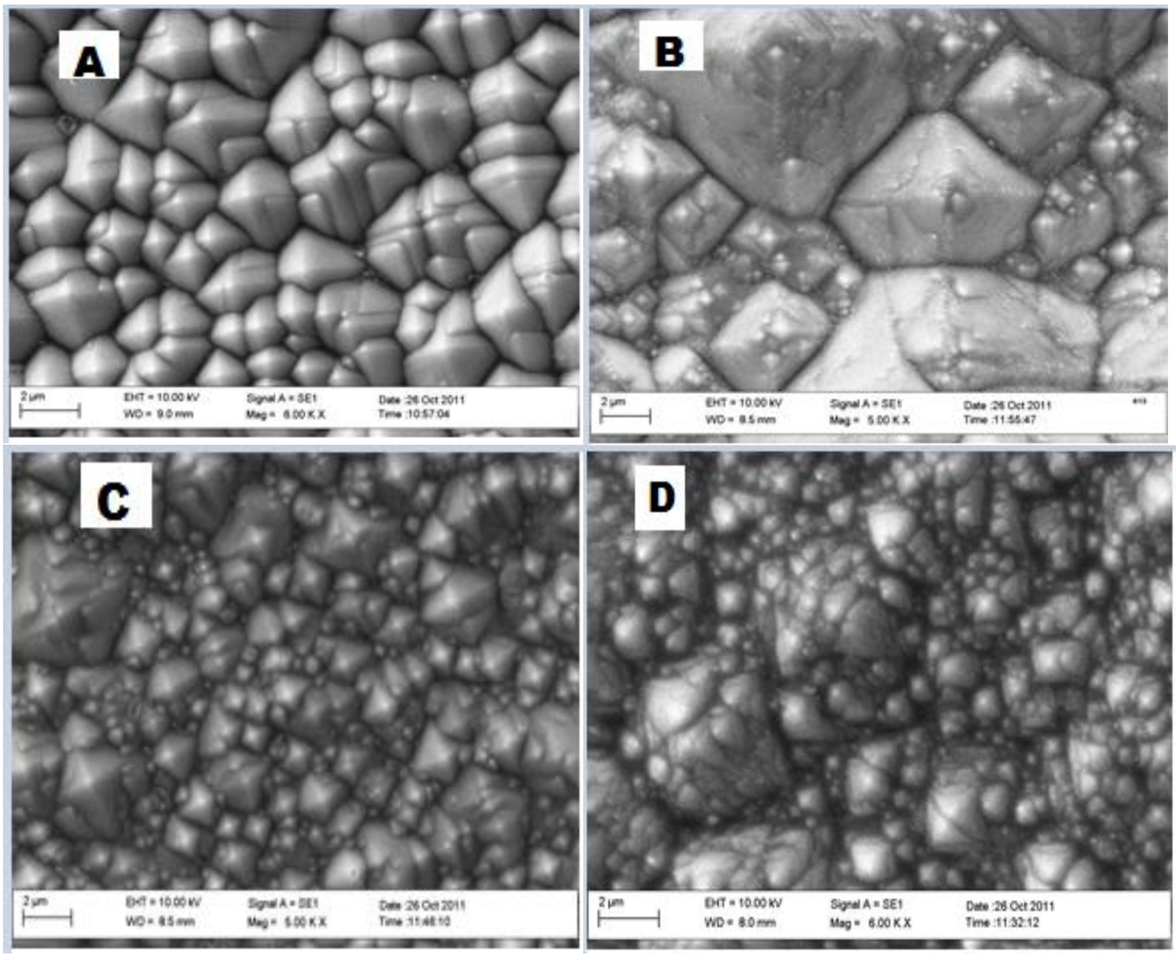


Figure 1. SEM textured image of silicon wafers at optimum etching time (A) 30 Mins (B) 40 Mins (C) 50 Mins (D) 60 Mins

The SEM images of the temperature-dependence nature of the etched silicon wafers are shown in Figure 2 for the various temperatures. It can be observed that the substrates were well-covered with films of different morphological structures. For 60 ^{O}C, the SEM micrograph shows rough and well-covered pyramidal images, which indicates the formation of a complete grain within the films. Increasing the deposition temperature to 70 ^{O}C, it was observed that the films were rougher with the formation of some compact grain-like features. The gradual formation of smaller grain-like features was due to the increased nucleation rate as the deposition temperature increased. Further increase in deposition temperature from 70 ^{O}C to 80 ^{O}C, increased the formation of the pyramidal grain sizes. The SEM images corroborate the results obtained by the surface profile studies. However, at 90 °C, the SEM image indicated surface damage showing that beyond 80 °C etching temperature, the etching process was destructive on the silicon wafers.

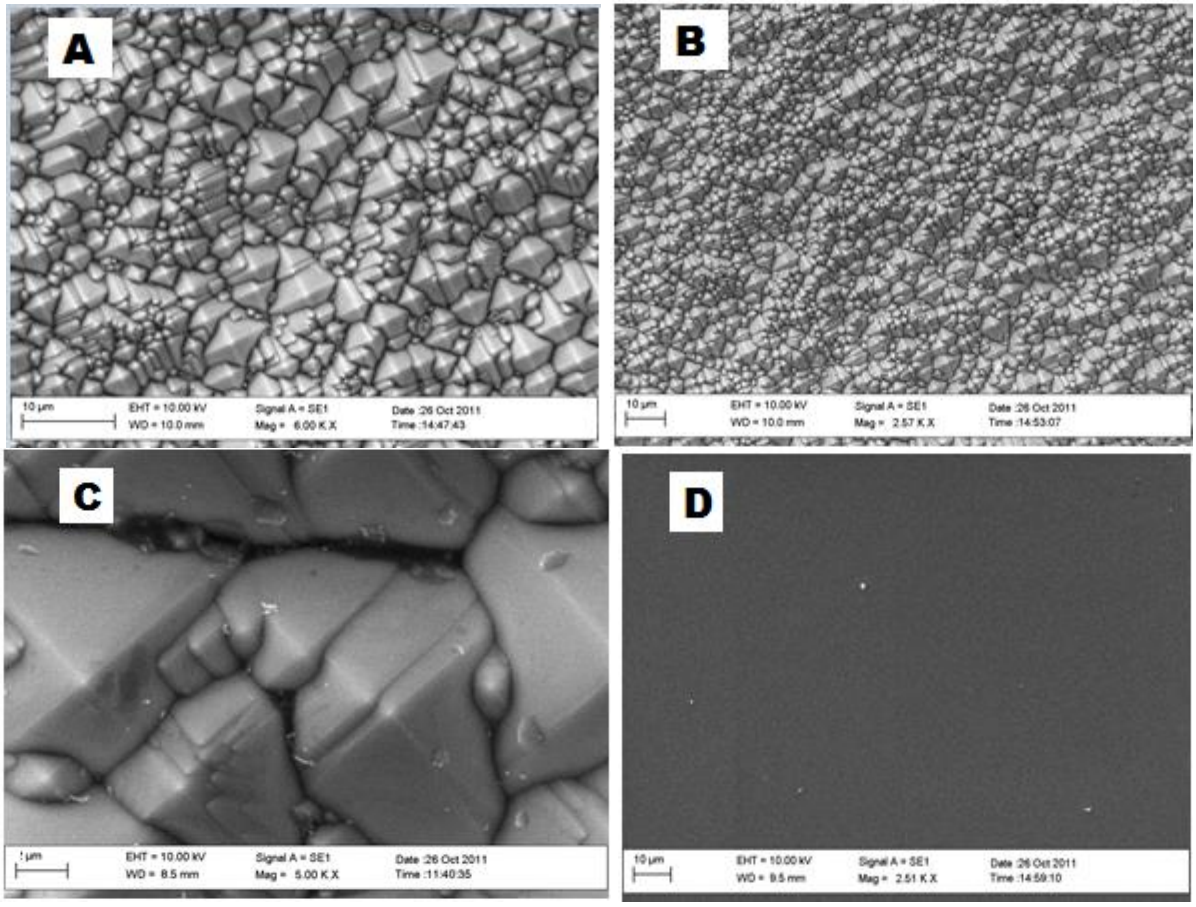


Figure 2. SEM textured images of silicon wafers at optimum temperature (A) 60^0 C (B) 70^0C (C) 80^0 C (D) 90^0 C

Figure 3 shows the SEM textured images of the surface morphologies at different concentrations for the silicon wafers. The pyramids produced were of different sizes when compared to that in Figure 1. Correspondingly, Park et al [24] have reported similar observations for crystalline silicon due to concentration modulations. Furthermore, the different morphological structures exhibited by the material may be the direct consequence of the variation in concentration and surface roughness exhibited by the material.

3.3 Effect of process parameters on the textured silicon wafers

Figure 4 shows the dependence of the reflectance of the textured wafers on the etching time. It was observed that the reflectance decreased as the etching time increases reaching an optimum value at 40 min etching time. Further increase in time beyond this optimum time has no significant effect on the reflectance as shown in Figure 4.

Figure 5 shows the relationship between the process temperatures and the optical reflectance. The optical reflectance was observed to decrease with increasing etching temperature as seen in the plot in Figure 5. As the temperature of the etching process was increased at an interval of 10 °C, there was a corresponding reflectance reduction of less than 5%. An indication that reduced optical reflectance can be achieved by increasing the process temperatures.

Figure 6 illustrates the effect of the etchant concentrations on the etched silicon wafers. The plots showed that the optical reflectance spectra of all the textured silicon wafers were less than 10%, suggesting an effective texturing process. Also from the plot, varying the IPA and KOH concentrations correspondingly increased the presence of the OH- group; therefore increasing the wettability of the mixture. This led to a decrease in optical reflectance in all the solution mixtures varied around the optimized mixture condition. Therefore, varying the etchant concentration reduces the overall surface reflectance of the textured wafer.

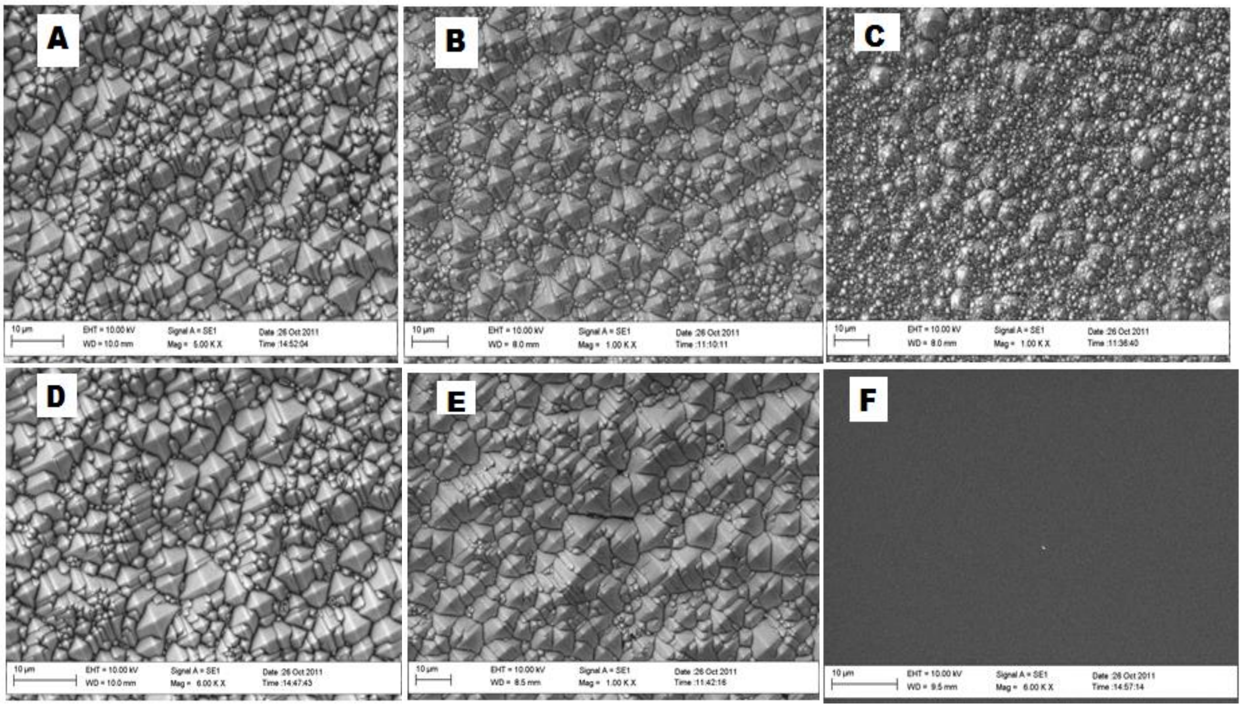


Figure 3. SEM textured images of silicon wafers at mixture concentrations (A) 1:2:46 (KOH:IPA:DI) (B) 1:2:46 (KOH:IPA:DI) (C) 2:4:46 (KOH:IPA:DI) (D) 2:4:46 (KOH:IPA:DI) (E) 4:4:46 (KOH:IPA:DI) (F) 4:4:46 (KOH:IPA:DI)

It is also noted that the reflectance increases with wavelength for all the samples and also with time and temperature. Generally, all the samples have low reflectance values that lie between 0 and 45% for textured silicon wafers at various temperatures and between 0 and 23% for textured silicon wafers at various etching times. However, at wavelengths greater than 500 nm, the material has the highest average values, which may be a result of the film density decreasing with the etching parameters. The values of the estimated reflectance are in accordance with the results of Fashina et al [11] on crystalline silicon. The reflective property of the material makes the sample a good material for anti-reflective coatings.

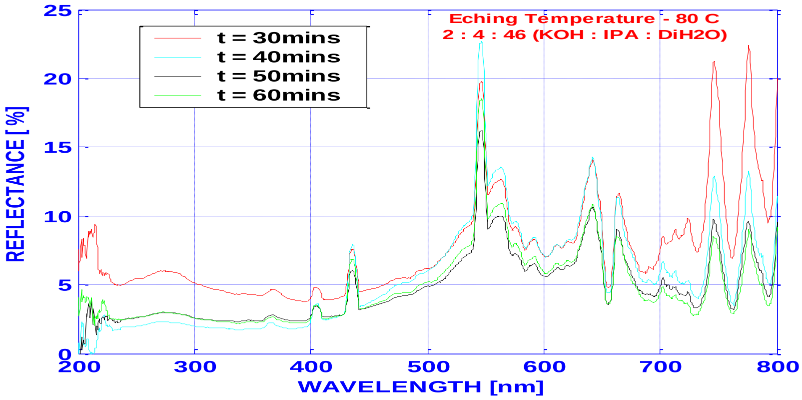


Figure 4 Reflectance spectral of textured silicon wafers with etching time

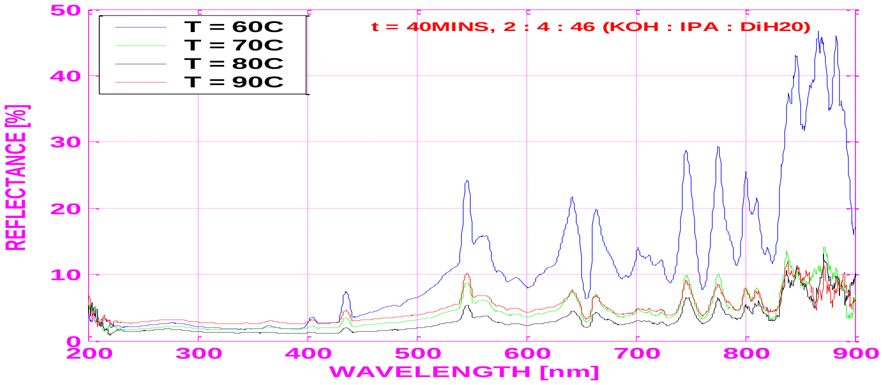
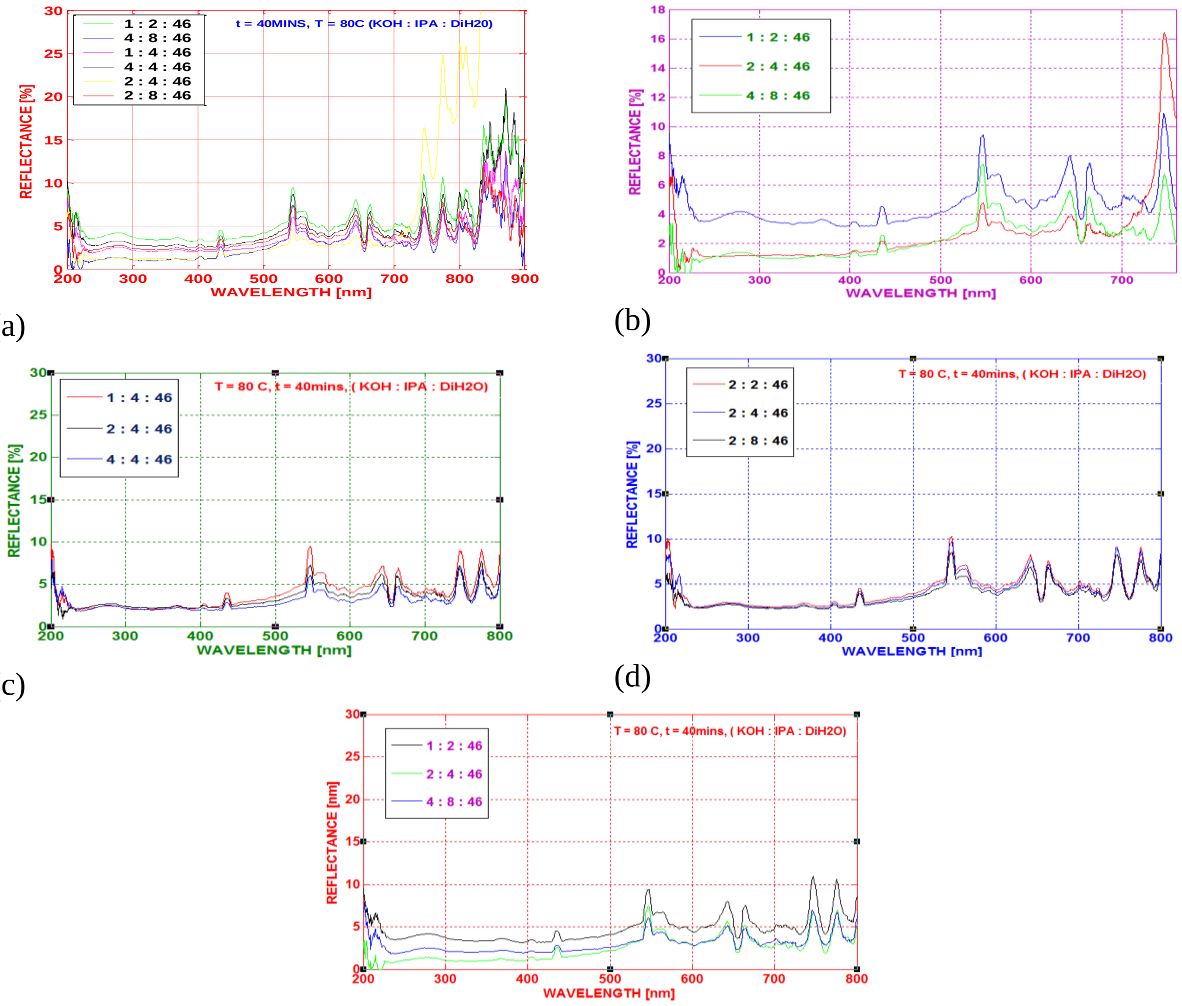


Figure 5: Reflectance spectral of textured silicon wafers with etching temperatures



(a)

(b)

(c)

(d)

(e)

Figure 6. Optical spectral reflectance of optimum concentration for textured silicon wafers at (a) various mixture concentrations (b) different KOH concentration (c) different IPA concentration (d) different KOH and IPA concentrations (e) different KOH and IPA concentrations

4. Conclusion

In this study, process variables have been identified such that the results show low-reflectance properties and surface textures which are compatible with optoelectronic devices as well as other solar cell fabrication processes. Minimizing the reflection loss from the front surface of a solar cell is the most important reason for surface texturing activities. Notwithstanding this salient reason, several other factors must be placed into consideration in ensuring that the texturing system is compatible with other solar cell production procedures and processes to achieve minimal cost implications. Furthermore, an operational surface texturing process at 80 °C and low concentrations of KOH and IPA was established. The etching process, using the solution of KOH: IPA produced pyramidal shapes of different dimensions. The average roughness of the etched silicon wafers was within a broad range of values of less than 10 % in all etched samples. The SEM analysis confirmed the pyramidal nature of the grains that are influenced by the surface roughness. The reflectance increased with wavelength as well as etching time and temperature indicating that the texturing technique has the potential to be adapted to solar cell fabrication. Thus, the texturing process applied to silicon wafers provided an enhanced surface structure in terms of morphology and reflectance which can be used in solar cell applications.

Nomenclature

SWs	Silicon wafers
KOH	Potassium hydroxide
IPA	Isopropyl alcohol
CZ	Crochralski
SC	Solar cell
NaOH	Sodium hydroxide
LiOH	Lithium hydroxide
CsOH	Cesium hydroxide
NH_4O	Ammonium hydroxide
TBA	Tertiary-butyl alcohol
DI	Deionized water
BOE	Buffered-oxide-etchant
HF	Hydrogen fluoride

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