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Passivation quality of a stoichiometric $\text{SiN}_x$ single passivation layer on crystalline silicon prepared by catalytic chemical vapor deposition (Cat-CVD) and successive annealing

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Abstract

A silicon nitride ($\text{SiN}_x$) single passivation layer, prepared by catalytic chemical vapor deposition (Cat-CVD) and successive annealing, shows high passivation quality on crystalline silicon (c-Si) wafers. Effective minority carrier lifetime ($\tau_{\text{eff}}$) monotonically increases with increase in deposition substrate temperature ($T_s$) for samples passivated by as-deposited $\text{SiN}_x$ films, while more significant increase in $\tau_{\text{eff}}$ by annealing tends to be seen for the samples with $\text{SiN}_x$ films deposited at lower $T_s$. The $\tau_{\text{eff}}$ obtained for the sample deposited at $T_s$ of 100 °C and pressure ($P$) of 10 Pa, after annealing at 350 °C for 30 min in
N$_2$, is about 3.0 ms, corresponding to a surface recombination velocity (SRV) of 5.0 cm/s. According to measured H content and fixed charge density ($Q_f$) in the SiN$_x$ films, $Q_f$ partly contributes to the passivation quality of the films particularly before annealing, while H content plays an important role on improving passivation quality of the films after annealing.
1. Introduction

An excellent coating layer on a single crystalline silicon (c-Si) surface is essential for producing high-efficiency c-Si solar cells. Such a coating layer needs high optical transparent property and high passivation quality for reducing the recombination of photo-generated carriers at c-Si surface [1]. Catalytic chemical vapor deposition (Cat-CVD), often also referred to as hot-wire CVD [2], is a method of depositing thin films by decomposing gas molecules on a heated catalyzing wire. Cat-CVD can realize plasma-damage-less deposition, and high-quality film/c-Si interface property is thus expected. We have so far demonstrated that silicon nitride (SiN$_x$)/amorphous silicon (a-Si) stacked passivation layers prepared by Cat-CVD on c-Si realize an extremely low surface recombination velocity (SRV) of < 1.5 cm/s [3]. However, parasitic absorption in the a-Si film may result in reducing c-Si solar cell efficiency. In order to overcome this problem, in previous study, we inserted Si-rich SiN$_x$ film instead of a-Si film. The obtained results show that SiN$_x$/Si-rich SiN$_x$ stacked layers as passivation films on c-Si achieves a SRV of as low as 3 cm/s with 30% improvement in transparency at a wavelength of 400 nm compared to that of SiN$_x$/a-Si films [4]. We have also found that annealing process and H content play important roles in improving the passivation quality of the stacked layers [5]. Ideally, further improvement in transparency should be achieved, because the Si-rich SiN$_x$ films still have considerable light absorption. Therefore, in this study, we study on the passivation quality of a Cat-CVD stoichiometric (refractive index of ~ 2) SiN$_x$ single layer on c-Si. SiN$_x$ films have
sufficiently high transparency and have been widely used as passivation and anti-reflective coating layers on c-Si [6-7]. The obtained results demonstrate that $\tau_{\text{eff}}$ is improved significantly by annealing. Samples prepared at lower substrate temperature during deposition ($T_s$) show more significant improvement in $\tau_{\text{eff}}$ by annealing, probably due to higher H content. Highest $\tau_{\text{eff}}$ obtained for the sample deposited at $T_s$ of 100 °C and pressure ($P$) of 10 Pa is about 3 ms, corresponding to SRV of 5.0 cm/s. Passivation quality of SiNx films on c-Si have been investigated by many researchers [6-17]. Most of SiNx films were prepared by plasma-enhanced CVD (PECVD) and low surface recombination velocities have been achieved. It has been reported that a low SRV of 2 cm/s can be achieved when PECVD SiNx films are deposited on 3-5 $\Omega$cm n-type Si wafer [11]. Schmidt et al. have achieved SRV lower than 10 cm/s for 1.5 $\Omega$cm p-type Si wafers passivated by stoichiometric SiNx films [14]. There are, however, few reports for the passivation of c-Si surface using Cat-CVD SiNx films [16, 17]. The work of Cat-CVD SiNx/c-Si was already published by our group in 2003 [17]. However, at that time, the effect of annealing and the role of H content in the films were not noticed, and the results obtained are not as good as those shown in this paper.

2. Experimental procedure

2.1. Sample preparation

After cleaning c-Si wafers in diluted (5%) hydro-fluoric acid (HF) solution to remove native oxide on c-Si surface, 100-nm-thick SiNx films were deposited by Cat-CVD. SiNx films with an approximately same refractive index of ~ 2 were deposited at various $T_s$
and $P$. SiN$_x$ films were also deposited onto glass substrates for optical transmission measurements. We also formed 100-nm-thick SiN$_x$ films on quartz substrates to measure defect density of the films. The deposition conditions of the SiN$_x$ films are summarized in Table I. To measure H content by Fourier-transform infrared spectroscopy (FTIR), SiN$_x$ films with a thickness of about 100 nm were deposited on c-Si substrates with a high resistivity of 3460 $\Omega$cm [18]. Fixed charge density ($Q_f$) of SiN$_x$ films was calculated based on the results of capacitance–voltage measurement for metal-insulator-semiconductor (MIS) structures [5, 19]. Firstly, a 100-nm-thick Aluminum (Al) layer was deposited on one side of 2 $\Omega$cm p-type floating-zone (FZ)-grown c-Si wafers by evaporation. The Al/c-Si structure was annealed at 400 °C for 15 min in N$_2$ atmosphere to obtain Ohmic contact. SiN$_x$ films were then deposited on the other surface of the c-Si wafers. Finally, 2-mm-diameter Al electrodes were evaporated on the SiN$_x$ layers through a hard mask. Some samples were annealed at 350 °C for 30 min before the evaporation of the circular Al electrodes in order to investigate the effect of annealing on $Q_f$. 100-nm-thick SiN$_x$ films were deposited on both sides of 290-$\mu$m-thick n-type (100) FZ Si wafers with a resistivity of 2.5 $\Omega$cm to measure effective minority carrier lifetime ($\tau_{eff}$). Samples were then annealing in N$_2$ atmosphere to investigate the effect of annealing on the passivation quality of SiN$_x$ films.

2.2. Characterization of prepared samples

The thickness and refractive index of all the samples were measured on J. A. Woollam, HS-190™ spectroscopic ellipsometer, using Cauchy model for data analysis [20].
The film density was measured by X-ray reflectivity [20]. Atomic contents of the SiN\textsubscript{x} films were measured by X-ray photoelectron spectroscopy (XPS). The transmission spectra of SiN\textsubscript{x} films were measured in a Shimadzu, UV-3150 ultraviolet-visible-near infrared spectrophotometer. Defect density was calculated by using electron spin resonance (ESR) [21]. In order to investigate the passivation quality of the SiN\textsubscript{x} layers on c-Si, we carried out microwave photo-conductivity decay (μ-PCD) measurement (Kobelco LTA-1510EP) using a 904 nm wavelength pulse laser with a photon density of 5×10\textsuperscript{13} cm\textsuperscript{-2} [22]. The method to determine SRV has been described in a previous paper [4].

3. Results and discussion

3.1. Passivation quality of SiN\textsubscript{x} films on c-Si wafers

The effect of annealing on the passivation quality of various SiN\textsubscript{x} films has also been reported by many authors [9, 23, 24]. It is demonstrated that the rearrangement of SiN\textsubscript{x} structure and H diffusion during annealing can terminated defects at SiN\textsubscript{x}/c-Si interface, thus, which results in improvement in passivation quality. In previous study, we also found that passivation quality of Cat-CVD SiN\textsubscript{x}/Si-rich SiN\textsubscript{x} films on c-Si wafer is enhanced significantly after annealing [4, 5]. In this study, we thus firstly investigate the effect of annealing temperature (T\textsubscript{a}) and annealing time (t\textsubscript{a}) on passivation quality of stoichiometric SiN\textsubscript{x} single films on c-Si wafers. In this experiment, stoichiometric SiN\textsubscript{x} films were deposited at T\textsubscript{s} of 50, 100, and 150 °C at a fixed P of 10 Pa. Figure 1(a) shows \(\tau_{\text{eff}}\) of SiN\textsubscript{x} films deposited at T\textsubscript{s} of 50, 100, and 150 °C at a fixed P of 10 Pa, and H concentration of those at a T\textsubscript{s} of 150 °C as functions of T\textsubscript{a} with a fixed duration of 30 min.
τ\text{eff} increases with increase in T\text{a}, reaches maximum value, and then, decreases dramatically. The improvement in τ\text{eff} on T\text{a} may be related to the diffusion of H atoms in the films and the termination of defect at SiN\text{x}/c-Si interface by H atoms during annealing. T\text{a} of 350 °C might be a proper temperature to support sufficient energy for H diffusion and defect-termination, resulting in the formation of good-quality SiN\text{x}/c-Si interface. Figure 1(b) shows FTIR spectra of SiN\text{x} films deposited at a T\text{s} of 150 °C at various T\text{a} for 30 min. Increase in H content after annealing at 350 °C, shown in the FTIR spectra, is due to increase in the number of Si-H bonds. This suggests that H atoms diffuse to a c-Si substrate and recombine with dangling bonds on c-Si surface, resulting in increase in Si-H bonds. However, at excessively high T\text{a}, H atoms can be released to environment and do not terminate defects, and τ\text{eff} is low [15-25]. As shown in Figs. 1(a) and 1(b), H concentration and Si-H bonding density decrease dramatically at T\text{a} of 450 °C, which is a clear evidence for H loss to environment.

Passivation quality of SiN\text{x} films can be stable for long time annealing, as shown in Fig. 1(c). Degradation of τ\text{eff} at very long t\text{a} may be due to H desorption caused by breaking of Si-H bonds and N-H bonds, when sample was annealed for very long time. In next steps, we used T\text{a} of 350 °C and t\text{a} of 30 min as ideal annealing conditions for all samples for high passivation quality improvement after annealing.

Figure 2 shows τ\text{eff} of c-Si wafers with SiN\text{x} films as functions of T\text{s} and P before and after annealing at a T\text{a} of 350 °C for 30 min. τ\text{eff} of as-deposited film before annealing increases with increase in T\text{s}, while decreases with increase in P. The low τ\text{eff} obtained at
low $T_s$ is considered to be due to low film quality or the effect of etching by atomic H, which is known to occur more significantly at low $T_s$ [26]. The effect of etching may be negligible, because at low $T_s$ with high deposition rate, rapid covering of c-Si surface with SiN$_x$ films can suppress H etching effect. However, films deposited with high deposition rate (at low $T_s$ and high $P$) might lead to the insufficient coverage of c-Si surface, resulting in decrease in $\tau_{\text{eff}}$. The reason of low $\tau_{\text{eff}}$ at these conditions can also be explained by low $Q_f$, which will be discussed later. Figure 2 also shows that $\tau_{\text{eff}}$ is improved significantly after annealing. For annealed samples, $\tau_{\text{eff}}$ reaches its highest value at $T_s$ of 100 °C, then it decreases with increase in $T_s$, while $\tau_{\text{eff}}$ increases with increase of $P$ and reaches a saturated value at $P \geq 10$ Pa. $\tau_{\text{eff}}$ obtained for the as-deposited sample at $T_s$ of 100 °C and $P$ of 10 Pa is about 0.1 ms, corresponding to a SRV of 144 cm/s. $\tau_{\text{eff}}$ of the same samples increases up to 3 ms after annealing, which corresponds to a SVR of 5.0 cm/s.

Figure 3 shows the optical transmission spectra of SiN$_x$ films deposited at $T_s$ of 150 °C and $P$ of 10 Pa before and after annealing at a $T_a$ of 350 °C for 30 min, simulated value of transmission of a SiN$_x$ film is also shown. The appearance of fringe in transmission spectra is probably due to the effect of interference. We also plotted a simulated transmission spectrum of a SiN$_x$ film evaluated by using Beer’s equation [27], in which absorption coefficient and film thickness obtained by analyzing spectroscopic ellipsometry data using by the Cauchy mode are used. The stoichiometric SiN$_x$ films show sufficiently high optical transmission even in short wavelength region, unlike a-Si or Si-rich SiN$_x$ films [3, 4]. Furthermore, the transparency of the films does not significantly change by annealing. The very small difference in the transparency in a short wavelength region
might be related to the slight change of network structures such as Si-H bonds by annealing. The improvement in the passivation quality of stoichiometric SiN$_x$ films without decreasing transparency after annealing is of great advantage for the application of the films in c-Si solar cell fabrication.

### 3.2. Role of H content and fixed charge density on passivation quality of films

In order to investigate the origin of the high passivation quality of SiN$_x$ films, we evaluated H content and $Q_f$ in the films [6, 12, 15]. Positive fixed charges send minority carriers (holes) far away from an n-type c-Si surface. They can therefore reduce the trapping probability of minority carriers (holes) at defects near c-Si surface. Another way to reduce recombination is the termination of defects by H atoms. Figure 4 shows SiN$_x$ film density and H concentration in SiN$_x$ films before and after annealing at a $T_a$ of 350 °C for 30 min as functions of $T_s$ and $P$. H concentration of SiN$_x$ films were determined from FTIR spectra, parts of which are shown in Fig. 5. H concentration of SiN$_x$ films decreases with increase in $T_s$, which is properly due to more enhanced H desorption during deposition at higher $T_s$. Higher $P$ leads to larger amount of H in SiN$_x$ films, while Si-H/N-H bond density ratio is kept almost constant. Compared to SiN$_x$ films deposited at higher $T_s$, the SiN$_x$ film deposited at a $T_s$ of 50 °C have a Si-H peak shifted to lower wavenumber. This might be related to the lower electro-negativity of backboned Si atoms for SiN$_x$ films with lower N content [28]. Si-H bonding increases much with decrease in $T_s$ and increase in $P$, while N-H bonding slightly tends to increase with increase in $T_s$ and $P$. Samples with high H content show more effective increase in $\tau_{\text{eff}}$ by annealing, and H may contribute to improvement in
$\tau_{\text{eff}}$. Sample prepared at $T_s < 100$ °C, exceptionally shows less significant improvement in $\tau_{\text{eff}}$ even with high H content. This may be due to low film density. During annealing, low-density materials would release H in the molecular form, while denser films would make H desorption slower [16, 28]. Here, SiN$_x$ film density is $\sim 2.1$ g/cm$^3$ for the sample deposited at $T_s$ of 50 °C, while it is more than $2.4$ g/cm$^3$ for sample deposited at $T_s \geq 100$ °C, as shown in Fig. 4. H atoms might thus be released to atmosphere during annealing and not contribute to passivation, resulting in a low $\tau_{\text{eff}}$. Samples formed at higher $T_s$ with much higher film density can suppress H desorption, and H atoms can significantly contribute to passivating defects on the c-Si surface. We also similarly explain improvement in $\tau_{\text{eff}}$ after annealing samples deposited at various $P$. Improvement in $\tau_{\text{eff}}$ may be related to H concentration and density of the films. Most of films deposited at various $P$ have sufficiently high film density and samples deposited at higher $P$ have higher H concentration, and passivation quality is improved more significantly after annealing for SiN$_x$ films deposited at high $P$.

Figure 6 shows N content and $Q_f$ of SiN$_x$ films as functions of $T_s$ and $P$ before and after annealing at a $T_a$ of 350 °C for 30 min. $Q_f$ increases with increase in $T_s$, and reaches highest value at a $T_s$ of 250 °C, then it slightly decreases at 300 °C. $Q_f$ of $7.5\times10^{11}$ cm$^{-2}$ is quite low for the SiN$_x$ films deposited at $T_s$ of 50 °C, which can be one of possible reasons to explain low $\tau_{\text{eff}}$ for sample deposited at low $T_s$ and high $P$, as shown in Fig. 2. N content increases with increasing $T_s$ or decreasing $P$. It may be correlative to film density, which is related to the migration of radicals on c-Si surface during deposition. The origin of fixed
charges is known to be Si-dangling bond defects whose configuration is $N_3^+ \equiv Si^+$ in SiN$_x$ films, generally called K$^+$ centers [15, 21, 29, 30]. From Fig. 6, we can also see that $Q_f$ is proportional to N content in films. It is in good agreement with the suggestion of the origin of fixed charges as mentioned above. Decrease in $Q_f$ at $T_s$ of 300 °C might be related to decrease in Si-dangling bonds in SiN$_x$ films when they were deposited at sufficiently high $T_s$.

Figure 7 shows the defect density of SiN$_x$ films as functions of $T_s$ and $P$ before and after annealing at a $T_a$ of 350 °C for 30 min. One can see lower defect density at higher $T_s$. One possible reason for this tendency is more enhanced migration of radicals on c-Si substrate during deposition at higher $T_s$. Dangling bonds in SiN$_x$ films is related to Si-dangling bonds back bonded to three N atoms, $N_3^+ \equiv Si^+$, generally called K$^0$ centers, which consist of an unpaired electron and are observable by ESR [12, 13]. The defect density of SiN$_x$ films deposited at low $T_s$ may thus be related to N content in the films. Defect density of the SiN$_x$ film deposited at low $T_s$ is relatively low, and higher $P$ leads to lower defect density, as shown in Fig. 7. These trends are same as those of N content in SiN$_x$ films. It has been reported, by Lelièvre et al., that H atoms can combine with Si-dangling bond defects (K$^+$ centers) during annealing, resulting in the formation of neutralized dangling bonds (K centers) [15]. Other group has also reported that K$^0$ centers will be converted to K$^+$ and K$^-$ centers by annealing [30]. In this study, defect density (K$^0$ centers) and fixed charge K$^+$ decreases significantly by annealing, as shown in Figs. 6 and 7. These are clear evidences of defect termination by H in SiN$_x$ films during annealing. We can guess that H
can terminate defects not only inside SiN$_x$ but also at SiN$_x$/c-Si interface during annealing, and $\tau_{\text{eff}}$ is improved significantly by annealing. The samples with lower film density and higher H content show small change in $Q_f$ and defect density by annealing, as shown in Figs. 6 and 7. This may be because denser films can prevent more hydrogen atoms from releasing to atmosphere during annealing, which results in more H atoms combine with Si-dangling bonds in SiN$_x$ films, and more decrease in $Q_f$ and defect density. This consideration cannot explain the change of defect density and $Q_f$ by annealing for samples deposited at high $T_s$. The difference of H content in the films might be related to this phenomenon.

Finally, we discuss the origin of remarkable high $\tau_{\text{eff}}$ of Si wafers passivated by SiNx films. The value of $Q_f$ on the order of $10^{12}$ cm$^{-2}$ is high enough to express field-effect passivation. Fixed charge in SiN$_x$ films can thus partially contribute to suppression in surface recombination. $Q_f$, however, decreases after annealing, while $\tau_{\text{eff}}$ is significantly improved by annealing. This fact indicates that not $Q_f$ but defect termination by H atoms mainly contributes to improvement in the passivation quality of SiN$_x$ films, which is consistent with our previous results [5].

4. Conclusions

Stoichiometric SiN$_x$ single films prepared by Cat-CVD have good passivation quality on c-Si wafers. Samples prepared at lower $T_s$ and high $P$ show more significant improvement in $\tau_{\text{eff}}$ by annealing. The possible reasons for this effect are the diffusion of H atoms in the films and the termination of defect at SiN$_x$/c-Si interface by H atoms during
annealing. H content in the films thus plays an important role on improving $\tau_{\text{eff}}$. The highest $\tau_{\text{eff}}$ obtained is 3 ms, corresponding to SRV of as low as of 5.0 cm/s. This study highlights the application of the Cat-CVD stoichiometric SiN$_x$ films as passivation layers for c-Si solar cells.

Acknowledgement

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References


Figure captions

Fig. 1. (a) $\tau_{\text{eff}}$ of SiN$_x$ films deposited at $T_s$ of 50, 100, and 150 °C at a fixed $P$ of 10 Pa, and H concentration of those at a $T_s$ of 150 °C as functions of $T_a$ with a fixed duration of 30 min. (b) FTIR spectra of SiN$_x$ films deposited at a $T_s$ of 150 °C at various $T_a$ for 30 min. (c) $\tau_{\text{eff}}$ as a function of $t_a$ at a $T_a$ of 350 °C.

Fig. 2. $\tau_{\text{eff}}$ as functions of $T_s$ and $P$ before and after annealing at a $T_a$ of 350 °C for 30 min in N$_2$ atmosphere.

Fig. 3. Transmission spectra of SiN$_x$ films deposited at $T_s$ of 150 °C and $P$ of 10 Pa before and after annealing at a $T_a$ of 350 °C for 30 min, simulated value of transmission of a SiN$_x$ film is also shown.

Fig. 4. Film density and H concentration of SiN$_x$ films before and after annealing at a $T_a$ of 350 °C for 30 min as functions of $T_s$ and $P$.

Fig. 5. FTIR spectra of SiN$_x$ films deposited at various $T_s$ and $P$.

Fig. 6. N content and $Q_f$ of SiN$_x$ films as functions of $T_s$ and $P$ before and after annealing at a $T_a$ of 350 °C for 30 min.

Fig. 7. Defect density of SiN$_x$ films as functions of $T_s$ and $P$ before and after annealing at a $T_a$ of 350 °C for 30 min.
Table I. Deposition conditions of SiN$_x$ films. Refractive index and thickness of deposited films are also summarized.

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Fig. 1

![Graph (a)](image1)

![Graph (b)](image2)
Fig. 2

(a) $P=10$ Pa

$\tau_{\text{err}}$ (ms) vs. $T_s$ (°C)

- ▲ Before annealing
- ● After annealing

(b) $T_s = 150°$ C

$\tau_{\text{err}}$ (ms) vs. $P$ (Pa)

- ▲ Before annealing
- ● After annealing
Fig. 3

![Graph showing transmission as a function of wavelength for SiNy/glass before and after annealing, and simulated transmission of SiNy film.](image)
Fig. 4

(a) $P = 10$ Pa

H concentration (cm$^{-3}$)

$T_s$ (°C)

Film density (g/cm$^3$)

- H concentration BA
- H concentration AA
- Film density

(b) $T_s = 150$ °C

H concentration (cm$^{-3}$)

$P$ (Pa)

Film density (g/cm$^3$)

- H concentration BA
- H concentration AA
- Film density
Fig. 5
Fig. 6

The upper graph shows the variation of $Q_r$ (cm$^{-2}$) with $T_s$ (°C) at $P = 10$ Pa. The data points represent:
- $Q_r$ before annealing (open squares).
- $Q_r$ after annealing (open circles).
- N content (%) (closed circles).

The lower graph illustrates the variation of $Q_r$ with $P$ (Pa) at $T_s = 150$ °C. The data points represent:
- $Q_r$ before annealing (open squares).
- $Q_r$ after annealing (open circles).
- N content (%) (closed circles).