Passivation Characteristics of New Silicon Oxide

Lei Gong, Chunlan Zhou, Junjie Zhu, Wenjing Wang

***Abstract*—Surface passivation on silicon is one of the hot topics for research in the field of silicon solar cells. Single layers of silicon oxide prepared by thermal oxidation, O_3 oxidation or chemical oxidation and the stack layer of silicon oxide combined with other dielectric layers as passivation layers have been widely used to reduce surface carrier recombination in silicon solar cells. This paper provides a new method for preparing silicon oxide that realizes an excellent passivation effect, in which perhydropolysilazane was used as a spin-coating precursor. The effective minority carrier lifetime for n-type silicon passivated by a SiO_x layer increases with SiO_x thickness and can reach 1 ms when thickness is up to 100 nm. Moreover, when SiO_x layers with a thickness of 100 nm or more were capped by an AlO_x or SiN_x layer, the effective minority carrier lifetime could get a value above 2 ms, which is much higher than the value for single SiN_x or AlO_x (approximately 500–750 μs). This may result from the high positive fixed charge in the SiO_x layer and high hydrogenation of silicon surface induced by H diffusion from AlO_x or SiN_x to SiO_x and stored in it.**

***Index Terms*—interface fixed charge, passivation, perhydropolysilazane, silicon oxide**

I. INTRODUCTION

At the silicon solar cell surface, due to the destruction of the crystal periodic structure, dangling bonds are generated, forming defect levels in the forbidden band, which seriously affect the minority carrier lifetime[1]. With the thickness of silicon solar cells continuing to decrease in order to save cost, the diffusion length of minority carriers may be close to or even larger than the thickness of the wafer; so, some carriers will diffuse to the surface to recombine, leading to surface passivation on silicon more important than before. Surface recombination is realized by a combination of the electric field effect and chemical components[2]. The fixed charge Q_f in the passivation film forms an electric field on the surface of the substrate, causing the energy band to bend and inhibit the movement of the minority carrier to the surface, thus leading to the field-effect passivation; however, the dangling bonds can be saturated by hydrogen atoms during the preparation and annealing of the passivation layers, reducing the interface state density D_{it} and increasing the minority carrier lifetime. SiNx, AlOx and SiOx are widely used as passivation materials [3-5]. Additionally, Si–SiO2 has a lattice-matched interface and positive Q_f to provide chemical passivation and field-effect passivation, respectively [6, 7]. Most of the silicon solar cells adopt a multi-layer film structure to ensure both the passivation and anti-reflection in practical applications.

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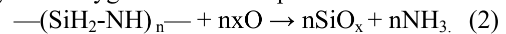
The most popular preparation methods of silicon oxide include thermal oxidation, wet chemical oxidation, plasma-enhanced chemical vapor deposition (PECVD), and plasma oxidation [8-10]. Thermal oxidation requires high temperature at 900–1200 °C; silicon atoms on the silicon surface are oxidized to SiO_2 in different atmospheres. However, the thermal oxidation requires large energy consumption, and the oxidation of the silicon wafer generates self-gap silicon atoms and form bulk defects. Wet chemical oxidation mainly includes H_2O_2, H_2SO_4, and HNO_3 oxidants[11]. The low growth rate of the wet chemical oxidation layer limits its application range, and using oxidants such as HNO_3 requires treatment of the waste, which increases the cost and could be harmful to the ecological environment. PECVD uses SiH_4 and N_2O or O_2 as a precursor to grow silicon oxide on the substrate, which requires a vacuum environment and a complicated process. In this paper, we use perhydropolysilazane (PHPS) as the spin-coating precursor to prepare a silicon oxide film, it is a low-temperature and uncomplicated process.

PHPS is mainly composed of Si–N and Si–H. It is mainly used for ceramic precursors, ceramic matrix composites and coating materials[12]. PHPS can be cured in an ultraviolet environment, and the –(SiH2–NH)– unit undergoes a hydrolysis reaction to form ammonia gas and $Si(OH)_4$ as an intermediate state[13]. During the subsequent heat treatment, portions of O and H overflow such that $Si(OH)_4$ forms a SiO_x grid. The principle of hydrolysis reaction is as follows[12]:

$$—(SiH_2–NH)_n— + 4nH_2O \rightarrow nSi(OH)_4 + 2nH_2 + nNH_3 \ . \ (1)$$

It can also be cured at appropriate temperature and form SiO_x directly with oxygen in the atmosphere:



$$—(SiH_2\text{-}NH)_n— + nxO \rightarrow nSiO_x + nNH_3 \quad (2)$$

Nagayoshi introduced a 100 nm SiO_x film which was made from the PHPS precursor into TiO_2 back reflector to promote the surface passivation on silicon[14], but that film is seldom used in solar cells. In this work, we explore the spin coating of a SiO_x film from a precursor of PHPS for surface passivation. We have studied the effect of annealing temperature and thickness of SiO_x films on the chemical component. In addition, the contribution of a single SiO_x layer on silicon surface passivation has been discussed. Finally, double layers of SiO_x/AlO_x:H, or SiN_x/SiO_x:H made by capping the SiO_x layer with plasma-enhanced atomic layer deposition (PE-ALD) AlO_x and PECVD SiN_x layers were investigated. The result shows that improvements were observed in the two stacks compared to the single passivation layer of SiO_x, AlO_x, and SiN_x.

II. Experiments

In this experiment, a silicon oxide film was formed by spin-coating a PHPS film on Si (100) substrates and using annealing at moderate temperature in atmosphere. An N-type double-sided polished c-Si substrate was adopted with resistivity of 1~10 $\Omega\cdot$cm. First, a wafer was immersed in H_2SO_4:H_2O_2 = 4:1 (volume ratio) at 85 °C for 10 min and rinsed in DI water; finally, the surface oxide layer was removed using 1% HF. After being dried in N_2, PHPS which was diluted with n-butyl ether, was double-sided coated on the silicon surface at 4500 r/min for 60 s by the spin-coating process. The as-coated film was baked at 150 °C for 3 min and then annealed in air atmosphere for 15 min in a box furnace. The annealing temperature was changed from 300 °C to 900 °C to obtain a single-layer SiO_x sample (as shown in Fig. 1 (a)); as for the structure of the stack layer, after depositing SiO_x film on the front and back surfaces of the silicon wafer and annealing, PE-ALD AlO_x with a thickness of 15 nm (as shown in Fig. 1(B1)) and an 80 nm PECVD SiN_x layer (Fig. 1(B2)) were capped on the SiO_x layers. Single AlO_x and SiN_x layers passivated silicon wafer were used as references. The SiO_x/AlO_x:H stack shown in Fig. 1(C) was obtained by exchanging the preparing order of AlO_x and SiO_x. After two-side deposition on silicon wafer, the single AlO_x, SiOx/AlOx:H, and SiO_x/AlO_x:H stacks were annealed at 400 °C for 10 min, and SiN_x layers, SiN_x/SiO_x:H, were annealed at 450 °C for 10 min in atmosphere. AlO_x layers were deposited at 200 °C with TMA and oxygen as the reaction precursor, and PECVD SiN_x layers were deposited at 400 °C with SiH_4 and NH_3 as the reaction precursor. The components and the thickness of the film were tested by Fourier transform infrared spectroscopy (FTIR) spectroscopy and ellipsometer, respectively. The effective minority carrier lifetime of a passivated silicon wafer and fixed charge in the dielectric film were measured through transient micro-photoconductive decay using WT-2000 instrument. The distribution of H in the film was tested by Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS).

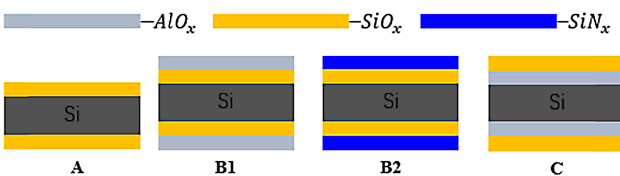


Fig. 1. Sample structures in the experiment; A is the structure of single SiOx-passivated Si wafer, B1 is a double-sided SiOx/AlOx:H stack-passivated Si wafer, B2 is a AlOx/SiOx:H stack-passivated Si wafer, and C is a SiOx/SiNx:H-passivated Si wafer.

III. Results and Discussions

A. Film components

The absorption FTIR spectra of the SiO_x film before and after annealing are shown in Fig. 2. This shows that the chemical component changes after annealing. The spectra for the as-deposited sample show the absorption peaks associated with the N–H peak (3,400 cm^{-1}), the Si-H peak (2,145 cm^{-1}), Si-O peak (1,080 cm^{-1}), and the Si-N peak (860 cm^{-1}). After annealing at 300 °C for 15 min, the N–H and Si–H peaks almost disappeared, whereas the Si–O peak intensity increased significantly, and the Si–N peak still appeared with lower intensity. As the annealing temperature increased, the intensity of the Si–O peak became higher, and the Si–N peak remained at a low level. This reveals that the Si–N, N–H, and Si–H bonds were broken, and the Si–O bond was formed by Si bonds reacting with the oxygen in the atmosphere during annealing.

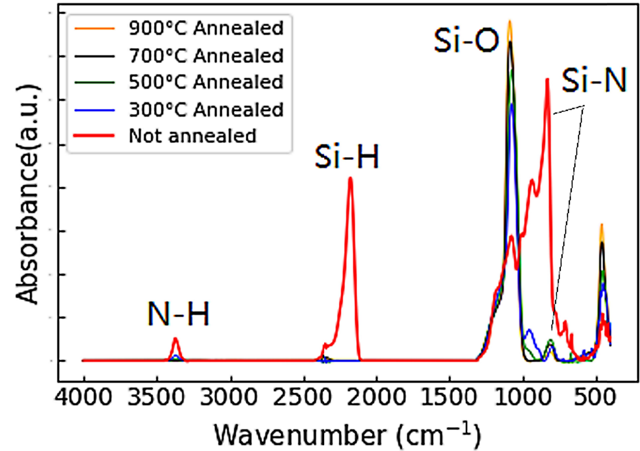


Fig. 2. The FTIR spectrum for SiO_x (280 nm) before and after annealing

Fig. 3 shows the peak of the Si–O normalized by Si–N peak (a) and the ratio of the Si–O peak's height to the Si-N peak's height (b) as a function of the annealing temperature. Their peak position moves toward a higher wavenumber with increasing annealing temperature; this indicates that the composition of the film is closer to chemical stoichiometry SiO_2. When the annealing temperature is up to 600 °C, the ratio of Si–O bond increases significantly, indicating that the Si–N bond more easily transfers to the Si–O bond under higher temperature. The PHPS film could be defined as SiO_xN_y:H, the atomic ratios of Si, O, and N can be calculated from the area of the Si–O and Si–N peaks in the FTIR spectrum. For the composition of SiO_xN_y, x and y satisfy the following relationship [15]:

$$x = \frac{[O]}{[Si]} = \frac{2[Si-O]}{[Si-O]+[Si-N]}$$

and

$$y = \frac{[N]}{[Si]} = \frac{4}{3}\cdot\frac{2[Si-N]}{[Si-O]+[Si-N]}.$$

$[Si-X]$ is the absolute concentration of the bond and can be calculated by $[Si-X] = K_{Si-X}\int_v \frac{\alpha(\omega)}{\omega} d\omega = K_{Si-X}A_{Si-X}$, the coefficient $K_{Si-O} = 1.5\times10^{19}\ cm^{-2}$, and $K_{Si-N} = 2.1\times10^{19}\ cm^{-2}$. After calculating the value of [O] / [Si] and [N] / [Si], we find that x continues to increase and y continues to decrease as the annealing temperature increases. The value of x is 1.95 at 300°C and 1.98 at 900°C, and that of y changes from 0.07 to 0.02. Therefore, the result reveals that N and H are released to the outside of the film in the form of H_2 and NH_3 during annealing, and the components of the film are mainly dominated by Si–O bonds. So, the film is gradually changed to the stoichiometric SiO_2 during annealing.

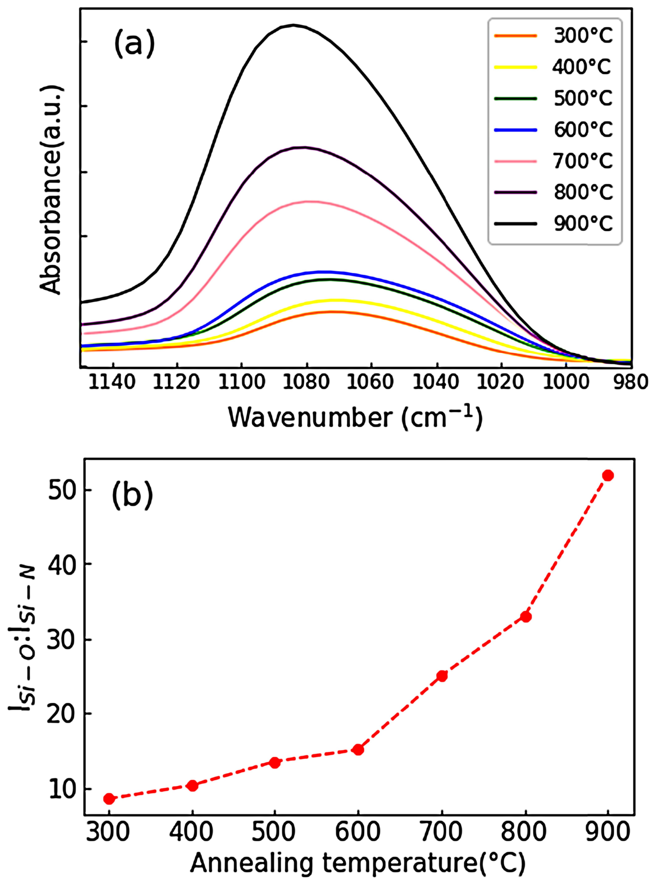


Fig. 3. FTIR spectrum of films annealed at different temperatures are normalized with the height of the Si–N peak and are shown in (a); the height ratio of Si-O and Si-N peaks varying with annealing temperature is shown in (b); the dash line is only for guidance.

B. Surface passivation quality

The effective minority carrier lifetime τ_{eff} for 280 nm SiO_x as a function of annealing temperature is shown in Fig. 4. In the low temperature range (200–500 °C), the value of τ_{eff} is quite small and rise slightly; then, τ_{eff} increases significantly to more than 1200 μs at 700 °C. However, τ_{eff} begins to decrease as the temperature further increases to 800 °C and above. To find the reasons for the decline, the corona charge test was performed on the films.

The corona charge method is a non-contact measurement method. By applying a charge of opposite polarity to the fixed charge Q_f to the surface of the sample, the field effect passivation is weakened, and the minority carrier lifetime is thus lowered; as the charge is continuously applied, the excess charge provides a passivation effect that causes τ_{eff} to begin to rise, forming a complete corona charge curve as shown in Fig. 5. The amount of charge applied at the lowest point of the curve is approximately regarded as the interface fixed charge Q_f, and the minimum effective minority carrier lifetime (τ_{min}) of the curve corresponds to the effect of chemical passivation, which is related to the interface state density D_{it}.

Fig. 7 shows that the fixed charge density is approximately $+1\sim2\times10^{12}\ cm^{-2}$, which is much higher than that in thermal oxidation of SiO_2 of $+3\times10^{11}\ cm^{-2}$[16]. The charge density gradually increases as the temperature increases, so the film annealed at higher temperature has better field effect passivation. As for the τ_{min}, it is approximately 1/10 of τ_{eff} for all the temperatures; so, the chemical passivation is very limited. The figure also shows that the relationship between τ_{min} and temperature is quite similar to that of τ_{eff} and temperature.

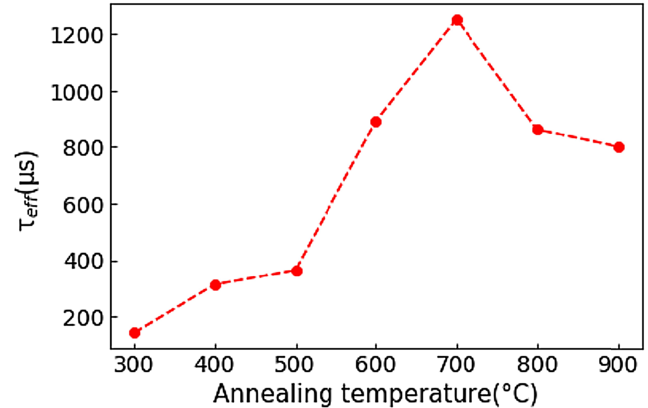


Fig. 4. The effective minority carrier lifetime of a 280 nm SiO_x varies with annealing temperature; the dash line is only for guidance.

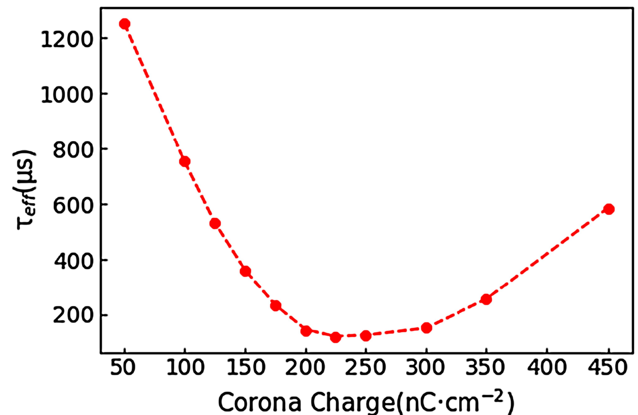


Fig 5. An example of the corona charge curve of 280 nm SiO_x film annealed at 700°C.

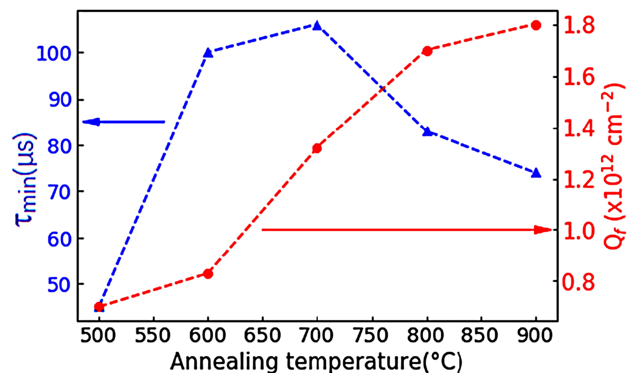


Fig 6. • τ_{min} and Q_f for the 280 nm single-layer SiO_x-passivated silicon wafer annealed at different temperatures; the dashed line is only for guidance.

Therefore, it can be concluded that the decrease of τ_{eff} at 800–900 °C mainly because of the increase of D_{it}. So we chose

the temperature of 700 °C as the annealing temperature for single SiO_x layer in the following study.

The relationship between film thickness and dilution ratio of PHPS with N-butyl ether is shown in Fig. 7. As the dilution ratio increases, the thickness of the film gradually decreases from 280 nm to less than 10 nm. The τ_{eff} as a function of the film thickness is plotted in Fig. 8. When the film is thinner than 15 nm, τ_{eff} is maintained at approximately 220 μs; then, τ_{eff} increases quickly as the film thickness increases and reaches 1100 μs at 80 nm. As the film thickness continuously increases, the upward trend of τ_{eff} becomes slow and reaches 1250 μs at 280 nm. It has been pointed out that when the lower thickness of the PHPS is thermal-treated, the film formation is less uniform, and cracks are likely to occur; this may be a cause of the serious decrease in the passivation effect[17].

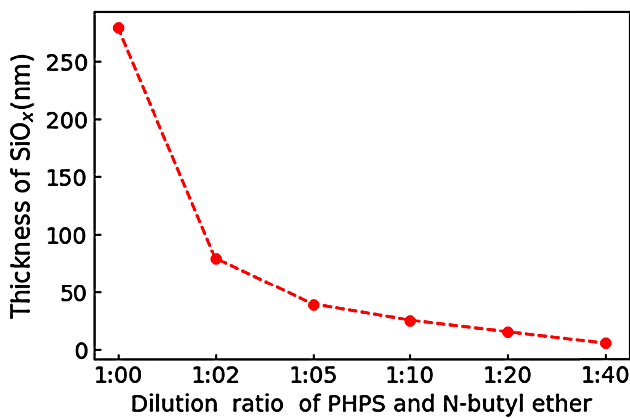


Fig. 7. The thickness of SiOx varies with dilution ratio after annealing at 700 °C for 15 min.

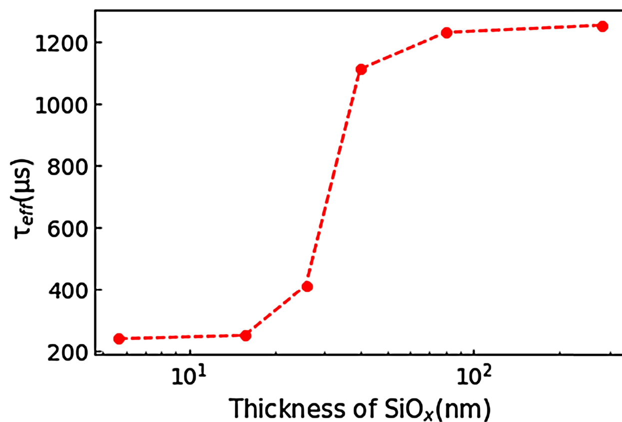


Fig 8. Effective minority carrier lifetime as a function of single SiOx layer's thickness after annealing at 700 °C for 15 min.

To investigate the passivation properties of the SiO_x film combining with other passivation layers, we prepared the stack layers of SiO_x/AlO_x:H and SiO_x/SiN_x:H, in which SiO_x is close to the silicon surface and AlO_x, SiN_x is the cap layer. It should be noted that for the stack layer, after studying the effect of annealing temperature of SiO_x layer on the surface passivation quality of SiO_x/AlO_x:H and SiO_x/SiN_x:H, we found the optimized annealing temperature of SiO_x to be 450 °C. Then, in the stacks preparation, the annealing of SiO_x was conducted at 450 °C. The SiO_x/AlO_x:H stack was prepared with different thicknesses of the SiO_x film, whereas for SiO_x/SiN_x:H, the thicknesses of SiO_x and SiN_x were fixed at 280 nm and 80 nm, respectively. The result for SiO_x/AlO_x:H is shown in the Fig. 9. The function of effective minority carrier lifetime with SiO_x thickness is basically the same as that of single SiO_x layer. It is evident that τ_{eff} values for SiO_x/AlO_x:H stack-passivated silicon wafers are higher than those for SiO_x; however, in the case of smaller thickness, the passivation effect is very limited, whether it is a SiO_x film (as shown in Fig. 8) or an SiO_x/AlO_x:H stack; for example, the SiO_x layer's thickness is smaller than 50 nm, and the stack layers have a poorer surface passivation effect compared with single AlO_x layer ($\tau_{eff} \approx 750$ μs). However, the τ_{eff} of the stack layer exceeds that of AlO_x as the SiO_x thickness is more than 50 nm; furthermore, it can reach more than 2200 μs when the thickness of SiO_x is 80 nm or more, whereas the value is approximately 1 ms for single 80 nm SiO_x. The interface fixed charge of the stack measured by a corona charge method is maintained at approximately $+3 \times 10^{11}\ cm^{-2}$ when the SiO_x thickness is above 80 nm. Compared with single SiO_x layer, Q_f of the SiO_x/AlO_x:H stack is significantly lower, but the τ_{min} of the stack is 10 times higher than that of the SiO_x film; this means that the chemical passivation is much stronger. As a result, the field effect passivation is relatively weakened, but with higher chemical passivation in the stack. By comparing the FTIR spectrum of the SiO_x film and SiO_x/AlO_x:H stack, we find that the FTIR of the SiO_x film after annealing at 450 °C does not show obvious peaks at 2200 cm^{-1} (Si–H), but the SiO_x/AlO_x:H stack does (not shown here). This may indicate that the diffusion of hydrogen from AlO_x films during annealing saturates the dangling bonds on the surface of the substrate to eliminate such recombination centers. To investigate the distribution of H in the stack, we used a TOF-SIMS test and got the spectrum of the SiO_x (80 nm)/AlO_x (15 nm) stack as shown in Fig. 10; a result of a 15nm AlO_x film is used as reference. In the single AlO_x film, the intensity of H^+ and SiH^+ remains at a low value and begins to increase until at the surface of Si. As for the stack of SiO_x/AlO_x:H, there are more H^+ or SiH^+ bonds in the SiO_x layer than in AlO_x layer, and an evident peak exists at the position closer to the substrate, even though the Si–H bonds in SiO_x have disappeared after annealing at 450 °C, which means that the H in AlO_x can diffuse into the SiO_x layer and accumulate at the surface of the Si substrate. This H saturates the dangling bonds to reduce D_{it} and achieve effective chemical passivation. In other words, this confirms that the excellent passivation of the SiO_x/AlO_x:H stack was mainly due to the enhancement of chemical passivation. This implies that higher concentration of H effusing from the AlO_x layer can be stored in the SiO_x layer, especially at the SiO_x/Si interface; these higher H concentrations provide better chemical passivation on silicon's surface than that passivated by single SiO_x and AlO_x layers. However, combining the TOF-SIMS results with the τ_{eff} result of the SiO_x/AlO_x:H stack shown in Fig. 9, it could be concluded that the effective effusion and storage H in SiO_x should require that the thickness of SiO_x is not less than 50 nm, and the thickness has less influence when the it closes 100 nm or more. It is interesting that when coating 80 nm SiO_x on 15 nm AlO_x and after annealing at 400 °C for 10 min, the minority carrier lifetime also increased from 750 μs for the AlO_x layer to about

1,200 μs for the stack. The Q_f values in AlO_x/SiO_x:H and AlO_x are identical; this means that reduced D_{it}, which results from the hydrogenation of the Si surface, contributes to the improvement of surface passivation on silicon.

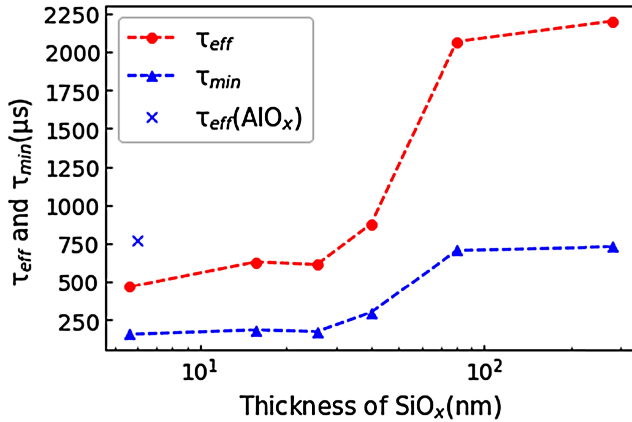


Fig. 9. τ_{eff} and τ_{min} of the SiOx/AlOx:H stack varies with thickness of SiOx. Before the deposition of 15 nm AlOx, SiOx were annealed at 450°C for 15 min. The stacks received extra annealing at 400 °C for 10 min. The τ_{eff} of 15 nm single-layer AlOx annealed at 400 °C for 10 min is the reference.

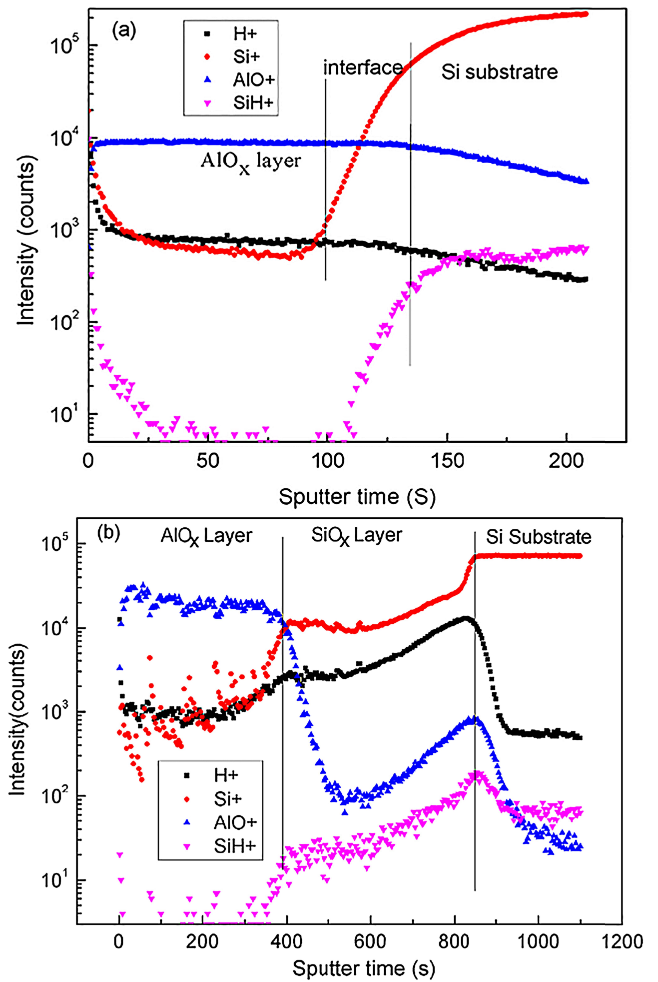


Fig. 10. TOF-SIMS spectra of single AlO_x layer (a) and the SiO_x/AlO_x:H stack (b), in which AlO_x is a 15 nm thick layer annealed at 400 °C for 10 min. Before deposition of AlO_x, 80 nm SiO_x was annealed at 450 °C. The stacks received extra annealing at 400 °C for 10 min. The boundaries of the passivation layer, the Si substrate and the interface are indicated by the vertical line.

As for the SiO_x/SiN_x:H stack, 280nm SiO_x was coated and annealed at 450°C for 15min; then, 80nm SiNx was then deposited by PECVD and then annealed at 450 °C for 10min. The result shows that the insertion of SiO_x between the substrate and SiN_x can increase the minority carrier lifetime from 422 μs (SiN_x) to 1,627 μs. The fixed charge density of the stack is approximately $+2\times10^{12}\ cm^{-2}$, which is nearly the same as that of SiO_x and less than that of SiN_x ($+3\times10^{12}\ cm^{-2}$). The τ_{min} of SiO_x/SiN_x:H stack is approximately 500 μs. The result of the corona charge measurement indicates that the field effect passivation of the SiO_x/SiN_x:H stack is substantially the same as that of the SiO_x film, and the chemical passivation effect is fairly substantial. This also confirms that SiO_x is the barrier of H effusion to the outside of stack layers. Therefore, the single SiO_x film has strong field-effect passivation provided by the Q_f, and its chemical passivation effect is limited. However, the SiO_x/AlO_x:H or SiO_x/SiN_x:H stack can effectively compensate for its disadvantage of chemical passivation.

IV. Conclusions

In conclusion, we have studied a new method for preparing silicon oxide by spin-coating PHPS film with some heat treatments. During PHPS films are annealed, the Si–O bonds are formed, and N and H are released to the outside to form a SiO_x film. The new SiO_x shows excellent passivation properties: the effective minority carrier lifetime increases with SiO_x thickness and can reach 1 ms when thickness is up to 100 nm. Moreover, when 100 nm or thicker SiO_x layers were capped by a AlO_x or SiN_x layer, the effective minority carrier lifetime could get the value above 2 ms, which is much higher than the value for single SiN_x or AlO_x (approximately 500–750 μs). The large density of positive fixed charge provides good field effect passivation and the diffusion of H from the AlO_x or SiN_x layer toward the silicon surface provides chemical passivation. The new SiO_x has quite a simple preparation process and requires low temperature compared with traditional ways. These advantages on preparation and performance provide good development potential and application prospects.

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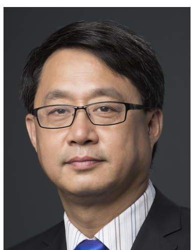

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