THERMAL STABILITY INVESTIGATIONS OF PECVD Al₂O₃ FILMS DISCUSSING A POSSIBILITY OF IMPROVING SURFACE PASSIVATION BY RE-HYDROGENATION AFTER HIGH TEMPERATURE PROCESSES

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ABSTRACT: This work investigates the changing passivation behavior of Al_2O_3 after different thermal treatments based on carrier lifetime (τ_{eff}), interface defect density (D_{it}) and fixed charge density (Q_{tot}) measurements. A concept of diffusing H species into the dehydrogenated Al_2O_3 films, termed as re-hydrogenation, has also been investigated for the PECVD deposited Al_2O_3 samples. Use of a-SiN_x:H as a capping layer as in Al_2O_3/a -SiN_x:H stack provides a better thermal stability for a thin PECVD Al_2O_3 layer. A comparison between single Al_2O_3 layer and Al_2O_3/a -SiN_x:H passivation stack after high temperature processes has been also performed in this work.

Keywords: Passivation, PECVD, defect density, electrical properties, re-hydrogenation.

1 INTRODUCTION

Al₂O₃ as a surface passivation layer for lowly and highly doped *p*-type *c*-Si is extensively researched for inclusion in the solar cell production sequence. In asdeposited state, Al2O3 provides nearly no surface passivation. An increase of minority charge carrier lifetime by 2-3 orders of magnitudes usually occurs after subsequent annealing, due to the activation of the passivation mechanism. This mechanism is partly due to an inherent high density of negative charges (Q_{tot}) in the layer (field-effect) and partly due to a lowered interface defect density (D_{it}) after annealing [1-3]. Though the passivation improves during the high temperature processes, the passivation level has been found to significantly degrade after a certain temperature and time period (>800 °C) [4]. This has been mostly attributed to the loss of H species from Al₂O₃ layer, thus leaving unsaturated dangling bonds acting as active recombination centers [1].

The current work investigates the changes in properties of Al2O3 layer after thermal processing at different temperatures. Effusion measurements for PECVD Al₂O₃ are used to investigate the role of H species in defining passivation quality of the layer at high temperatures. With an increasing temperature, gradual effusion of H species from the Al₂O₃ layer occurs leaving behind a large number of interface defect states. Changes in effective lifetime (τ_{eff}), interface defect density (D_{it}) and fixed charges (Q_{tot}) are used to explain the changing electrical properties of the layer with temperature. In this work, a new concept of re-hydrogenation is investigated in order to check the possibility of diffusing H species into the Al₂O₃/Si interface and revive the deteriorated passivation quality of the de-hydrogenated samples. This investigation provides deep insight about the effect on Al₂O₃/Si laver system due to a varying amount of H species in the Al₂O₃ layer. In the last section, the thermal stability of PECVD Al₂O₃/a-SiN_x:H stack has been investigated based upon carrier lifetime measurements performed after annealing processes performed at different temperatures.

2 THERMAL INVESTIGATION OF PECVD Al₂O₃

2.1 Experimental

Shiny etched surface float zone (FZ), low resistivity (1 Ω cm) *p*-type crystalline silicon (*c*-Si) wafers with a thickness of 250 µm and (100) crystal orientation were used for the investigations. Before the PECVD Al₂O₃ deposition process, the wafers were cleaned in a hot HNO₃ bath followed by a short HF dip. The final HF dip is applied to remove both the native oxide and the grown oxide (formed as a result of the HNO₃ bath) on top of the Si substrate. An inline industrial PECVD machine (Roth & Rau SiNA) was used to deposit Al₂O₃ films as well as a-SiN_x:H films on the *c*-Si samples.

2.2 The role of H species in passivation mechanism

Hydrogen species are believed to play an important role in defect passivation at the Al₂O₃/Si interface. Effusion experiments in cooperation with FZ Juelich were carried out to monitor the H species effusing out of the PECVD Al₂O₃ sample with an increasing temperature. For that, the sample was heated up inside a high vacuum quartz tube up to high temperatures with a defined heating rate of 20 K/min [6]. The effused gases from the sample were analyzed by a quadrupole mass spectrometer (QMA) to detect different mass-to-charge (m/z) ratios. For QMA used in H effusion measurements, the electrons generated by a heated filament are accelerated by applying a voltage of 50-100 V to cause ionization, and under such conditions, z is typically 1 [5].

Effusion signals for H species were detected in the form of mass-to-charge ratio (m/z) of 18, 17 and 2. Neglecting the presence of deuterium, these m/z values also feature in the cracking pattern of water molecule [6]. Overall effusion intensity of H₂ species (m/z=2) detected was measured by the effusion set up. Comparing the effusion signal of H species for PECVD Al₂O₃ to that of ALD Al₂O₃ layers, only small differences have been detected [7]. This also suggests that the thermal effusion mechanism of H species from the Al₂O₃ layer is virtually independent of these two deposition techniques.



Figure 1: Effusion intensity profile of H species for 100 nm PECVD Al_2O_3 layer over temperature. The m/z = 2 represents the overall H_2 detected during the measurement.

As shown in Figure 1, the effusion of H species starts already at low temperature of 250-300 °C and follows till a very high temperature (T>1000 °C). Up to the temperature of ~650-700 °C, the H₂ (m/z=2) effusion signal resembles (neglecting the spike at ~500 °C), the effusion curves of m/z=15 and m/z=17, 18 attributed to hydrocarbons and water respectively, which are likely to be present in the PECVD Al₂O₃ layer. Hence, it is likely that the effusion of H species at temperatures lower than ~700 °C is preferentially originating due to the surface and bulk desorption of water (H₂O, OH) and thermal cracking of hydrocarbons (CxHy). Peak for H₂ and increasing slopes for C_xH_y and OH, H₂O were observed at ~500°C. It should be noted that a dramatic improvement of the surface passivation has been reported for Al₂O₃ layers after annealing at the temperature of ~400-500 °C [4]. Hence, the H₂ effusion peak at this temperature could possibly be related to the effective diffusion of H species to the Al₂O₃/Si interface in order to activate the interface defect passivation mechanism. These conclusions, however, cannot be drawn only based upon effusion measurements¹. After 800 °C, a strong peak for the overall H₂ signal was observed in parallel to the decreasing slopes for H₂O and C_xH_y. This strongly implies that most of the H species that are effused from the sample are being effectively transported from the Al₂O₃/Si interface in a de-hydrogenation process. It is likely that such a de-hydrogenation process is forcing the thermal breakage of Si-H bonds leading to an immediate formation of stable molecules such as H₂ and H₂O effusing out of the sample.

2.3 Electrical characteristics of Al₂O₃ film

20 nm PECVD Al_2O_3 was deposited on both sides of the *c*-Si samples prepared as explained in section 2.1. After the diffusion process, the samples were annealed in N_2 ambient at different temperatures in a tube furnace. These samples were then exposed to the H_2 plasma atmosphere in a remote plasma hydrogen passivation (RPHP) system with a chamber temperature of ~450 °C for a substantially long time (45 min). Minority charge carrier lifetime (τ_{eff}), interface defect density (D_{it}) and fixed charge density (Q_{tot}) measurements were taken as the parameters for determining the passivation quality of the samples. τ_{eff} is measured using Sinton Consulting WCT-120 lifetime tester operated in generalized $(\tau_{eff} < 200 \ \mu s)$ or transient mode $(\tau_{eff} > 200 \ \mu s)$ [8, 9]. Minority charge carrier lifetime was measured for the symmetrical samples assuming infinite bulk lifetime of FZ wafers. Corona oxide characterization of semiconductors (COCOS) metrology was used to deduct the D_{it} and Q_{tot} values of the passivation layer using a CFORS tool from SDI Semilab.



Figure 2: Effective carrier lifetime (τ_{eff}) as a function of annealing temperature. Samples were first annealed at different temperatures and then subsequently exposed to the H₂ plasma atmosphere. The dotted lines serve only as guidelines for the eye. Samples were annealed either for 15 min (T<700 °C) or for 1 min (T>700 °C) before H₂ plasma exposure.



Figure 3: D_{it} and Q_{tot} values obtained as a function of annealing temperature. The uncertainty of the measurement is around $5 \times 10^{10} \text{ eV}^{-1} \text{ cm}^{-2}$ for D_{it} and $5 \times 10^{10} \text{ cm}^{-2}$ for Q_{tot} . Samples were annealed either for 15 min (T<700 °C) or for 1 min (T>700 °C) before H₂ plasma exposure.

Figure 2 and Figure 3 plot the changes in electrical parameters of the Al_2O_3 after subsequent annealing. The as-deposited samples showed low carrier lifetime values

¹ The heating rate of effusion experiment is much lower as compared to that used in standard thermal processing of solar cells. Thus the physical mechanism of defect passivation cannot be exactly correlated for these two processes.

(τ_{eff} <50 µs). The samples, which were annealed at lower temperatures of 350 °C and 425 °C, showed significantly low D_{it} and high Q_{tot} values. As expected, τ_{eff} increased to a value of 632 µs at 350 °C and 1900 µs at 425 °C after 15 minutes of annealing. A low D_{it} and an improved value of τ_{eff} indicate the gradual diffusion of H species to the Al₂O₃/Si interface at these temperatures. Annealing at the temperature of 600 °C resulted in slightly higher D_{it} and lower Q_{tot} values. The reason for higher D_{it} is possibly again related to the loss of H species from the Al₂O₃/Si interface at this temperature. Due to a very small change in D_{it} as compared to the D_{it} value observed at 425 °C, the sample however still showed a high charge carrier lifetime (τ_{eff} ~800 µs). At higher temperatures, significant changes in D_{it} and Q_{tot} values were observed. The sample annealed at 800 °C showed a higher value of D_{it} and a lower Q_{tot} value than samples that were annealed at lower temperatures. These changes are consistent with a significantly lower lifetime (τ_{eff} -232 µs) for this sample. The sample annealed at 1000 °C showed a very high D_{it} (~10¹² eV⁻¹cm⁻²), which is in the range equivalent to that of an as-deposited Al₂O₃ sample. This is consistent with a very low lifetime (τ_{eff} ~1 µs) obtained for this sample. It is likely that a nearly complete loss of H species from the Al₂O₃/Si interface is responsible for the deteriorated passivation for this sample. A very high Q_{tot} at 1000 °C could not be explained conclusively. It is possible that the undergoing structural changes in the Al₂O₃ layer at very high temperatures could be responsible for such a behavior [10].

2.4 Re-hydrogenation possibility of Al₂O₃ films

The previous sections underlined that the dehydrogenation process at high temperature processes deteriorates the passivation quality of Al₂O₃. The dehydrogenation of Al₂O₃ films was reported as an irreversible process, accredited to the structural changes in the layer at higher temperatures [7]. We studied the possibility of a re-hydrogenation process, which can diffuse H species into the Al₂O₃/Si interface of the dehydrogenated samples and possibly can revive the passivation quality of Al₂O₃ layers. The c-Si samples deposited with 20 nm PECVD Al₂O₃ were pre-annealed at different annealing temperatures as discussed in section 2.3. The samples were then exposed to the remote plasma hydrogen passivation (RPHP) system for a substantially long time of 45 minutes in order to facilitate the diffusion of H species into the sample. A detailed description of the remote plasma hydrogen passivation process can be found elsewhere [11].

After the subsequent H₂ plasma exposure, a loss of more than one order of magnitude in carrier lifetime was observed for the samples that were annealed at 350 °C and 425 °C. These samples also suffered a subsequent enhancement in D_{it} after H₂ plasma exposure. It should be noted that these samples had an excellent passivation quality before H₂ plasma exposure. This loss in minority charge carrier lifetime can be attributed to a phenomenon called 'Blistering', which has been observed in these samples after H₂ plasma exposure. Blistering is regarded as the formation of gaseous bubbles in or at Al₂O₃ films during a high temperature step, which can cause a significant loss in the passivation quality. The exact mechanism of blistering is still unknown, however, it is mostly attributed to an excess of H species present in the passivation layer and is more commonly observed on thicker Al₂O₃ passivation layers [12].

Figure 4 shows the scanning electron microscopic (SEM) image of the samples after successive hydrogenation. The black spots on the sample surface indicate the blistering phenomena. The samples, which were annealed at 350 °C and 425 °C, showed heavy blistering after H₂ plasma exposure. In contrast, the rest of the samples that were annealed at higher temperatures (600 °C, 800 °C and 1000 °C), showed no signs of blistering after H₂ plasma exposure.



Figure 4: Blisters as big as \sim 53 µm were observed in SEM images after H₂ plasma treatment of the samples that were previously annealed at 350 °C and 425 °C. The dark spots on the sample surface are blistering.

After H₂ plasma exposure, positive result was obtained for the sample that was previously annealed at a temperature of 800 °C for 1 minute. The minority charge carrier lifetime increased from 230 µs to 530 µs after the H₂ plasma exposure. Such a re-hydrogenation process was not reported before for Al₂O₃ passivated samples. The results re-assert that the annealing temperature and amount of H species present in the passivation layer plays a significant role in the passivation mechanism. The statement is also supported by an eventual reduction of D_{it} from 2.57x10¹¹ eV⁻¹cm⁻² to 1.87x10¹¹ eV⁻¹cm⁻² of the sample after the re-hydrogenation process. This sample also showed a slight reduction of D_{it} and nearly no change in the fixed charge density (Q_{tot}).

Photoluminescence (PL) measurements were performed for these samples before and after the H₂ plasma exposure of the samples using a 790 nm laser source. PL signal provides a direct measure of the passivation level based upon the change of minority charge carrier density due to the illumination of the wafer during the measurement [13]. Figure 5 shows the samples arranged in the order of annealing temperature: 350 °C, 425 °C, 600 °C and 800 °C. After an exposure to H₂ plasma, τ_{eff} strongly deteriorates for the samples annealed at 325 °C and 425 °C resulting in a very weak luminescence signal, not visible in the relative scale of the image (see Figure 5, right). The sample annealed at 600 °C is not much affected by the H_2 plasma exposure. The sample that was previously annealed at 800 °C, showed significantly improved PL signal after H₂ plasma exposure.



Figure 5: Photoluminescence measurements of the samples before (left) and after (right) the H₂ plasma exposure. The PL signal was calibrated with QSSPC transient method lifetime values at a specified carrier density of 10^{15} cm⁻³. After H₂ plasma, the lifetime values of the samples annealed at 350 °C and 425 °C was so low that they appear black in the image.

2.5 Discussion of the results

Samples which were thermally treated at 350 °C and 425 °C for 15 minutes showed an excellent passivation quality on c-Si material due to an activation of the Al₂O₃ passivation mechanism. For these samples, the drastic loss of passivation quality after H₂ plasma exposure can be attributed to an already high concentration of H within the samples prior to the exposure. Since the interface defects are already passivated during the annealing process, H species that are diffused by H₂ plasma exposure possibly causes simultaneous rupture of Si-H bonds at the interface, followed by the formation of volatile H₂. The formation of H₂ eventually increases the density of defect states at the Al2O3/Si interface and is possibly also responsible for the formation of blisters on the surface of the samples. No blistering was observed on the samples, which were annealed at 600 °C, 800 °C and 1000 °C before H₂ plasma exposure. These films are likely to be lacking in H species at the Al₂O₃/Si interface after thermal treatment at these temperatures, as suggested by high D_{it} values. This can be attributed to the effusion of H species at such high temperatures. Out of these samples, only the sample, which was thermally treated at 800 °C for 1 min showed a significant improvement in passivation, thus asserting a big role of temperature in determining the passivation quality of Al₂O₃ films. For this sample, large numbers of defect levels created due to the effusion of H species at high anneal temperatures are possibly compensated by the diffusion of H species into the Al₂O₃/Si interface. Thus a loss in passivation quality of the dielectric film after undergoing a high temperature annealing process seems to be partly compensated by an additional rehydrogenation step.

3 THERMAL STABILITY OF Al_2O_3/a -SiN_x:H STACK FOR REAR SIDE PASSIVATION

PECVD Al₂O₃ (10-15 nm) as the rear passivation layer is usually stacked with thicker capping layer of a-SiN_x:H (>80 nm). The thicker a-SiN_x:H layer not only protects the thin Al₂O₃ layer during further cell process steps, for example against metallization, but also provides better stability against higher thermal processes. To compare the thermal stability of the single Al₂O₃ layer and Al₂O₃/a-SiN_x:H passivation stack, minority charge carrier lifetime experiments were performed. The samples were prepared as detailed is section 2.1. Afterwards, 20 nm PECVD Al₂O₃ was deposited symmetrically on the samples. Passivation stacks were prepared by symmetrically depositing 15 nm PECVD Al₂O₃ layer followed by 80 nm PECVD a-SiN_x:H layer as capping layer. The samples were then annealed at different temperatures ranging from 350 °C to 1000 °C. τ_{eff} was measured for the samples after the annealing step. Assuming an infinite bulk lifetime of FZ wafers used for the experiments, the maximum value of surface recombination velocity ($S_{eff,max}$) was calculated for the symmetrical samples by using the equation [14]

$$S_{eff,\text{max}} = \frac{W}{2 \times \tau_{eff}}$$

where, W is the wafer thickness and τ_{eff} is the effective lifetime obtained from quasi steady state photo-conductance decay (QSSPC) method.



Figure 6: Upper limit of the surface recombination velocity ($S_{eff,max}$) plotted vs. annealing temperature. For the annealing temperature of 1000 °C, both samples showed very high maximum surface recombination velocity ($S_{eff,max}$ >1000 cms⁻¹).

Figure 6 plots the upper limit of S_{eff} for the samples that were annealed at different annealing temperature and times. It was observed that the Al₂O₃/a-SiN_x:H stack has better thermal stability compared to a thin Al₂O₃ layer, especially with increasing annealing temperatures. At 800°C, use of Al₂O₂/a-SiN_x:H as the passivation stack was observed to provide much better surface passivation $(S_{eff} < 15 \text{ cms}^{-1})$ as compared to that of single Al_2O_3 layer $(S_{eff} > 50 \text{ cms}^{-1})$. This can be accredited partly to the presence of PECVD a-SiNx:H that possibly blocks the tremendous effusion of H species from Al₂O₃/Si interface at the temperature T>700 °C (see Figure 1). Such a H diffusion barrier property of a-SiNx:H layer has also been discussed in the literature before [15]. Additionally, the better thermal stability of PECVD Al₂O₃/a-SiN_x:H stack is accredited to a high amount of H species present in the a-SiN_x:H layer that possibly diffuse into the Al₂O₃/Si interface and perform interface defect passivation.

The sample that was annealed at a high temperature of 1000 °C for 1 minute was observed to have a very high value of S_{eff} . This loss of passivation at a very high temperature of 1000 °C can be partly accredited to the overall de-passivation of the Al₂O₃/Si interface defects

due to the effusion of H species from the Al_2O_3/a -SiN_x:H stack at such a high temperature. Additionally, such higher temperatures are supposed to promote irreversible structural changes in the passivation layers that can deteriorate the passivation quality of the passivation stack [10].

4. SUMMARY

In this paper, we performed a detailed investigation of the thermal stability of Al₂O₃ layer on *c*-Si surfaces. We observed that a gradual effusion of H species occurs at all temperatures. Huge peaks of H species at higher temperature (T>700 °C) were attributed to the loss of H from the Al₂O₃ bulk and also from the Al₂O₃/Si interface, thus increasing the interface defect density of the layer. Such a high temperature was also found detrimental to the electrical properties of the PECVD layer. τ_{eff} , D_{it} and Q_{tot} measurements performed after thermal processes at different temperatures suggested a deteriorating passivation quality of the layer at temperature T>700 °C. This loss of passivation closely corresponds to the dehydrogenation of the Al₂O₃/Si interface in this temperature range. For such de-hydrogenated samples, we proposed a new concept of enhancing passivation level by exposing the samples in the H₂ plasma atmosphere. Such a process has been termed as rehydrogenation. A significant increment of τ_{eff} and PL signal showed that re-hydrogenation is possible for dehydrogenated Al₂O₃ samples and should be further investigated. Annealing of Al2O3 samples at lower temperatures (T~350-450 °C) showed heavy blistering and subsequent loss of passivation after the H₂ plasma exposure. This loss of passivation has been accredited to the excessive amount of H species present within these samples after H₂ plasma exposure. The H species diffusing from H₂ plasma atmosphere to the Al₂O₃/Si interface possibly interacts with the H species at the interface and forms volatile H2. Accumulation of H2 at the passivation interface causes lift-off effects reducing c-Si surface passivation and is likely to be responsible for the formation of blisters. The results also indicate that an excessive amount of H species in Al₂O₃ layer is detrimental for its passivation quality to the c-Si surface. Finally, we showed that the PECVD Al₂O₃/a-SiN_x:H stack has higher thermal stability as compared to that of single PECVD Al₂O₃ layer at higher temperatures (T>700 °C). The results presented in this work are supposed to have significance in understanding of the thermal processing of thin passivation layers.

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