Improved Process Stability on an Extremely Thin Amorphous/Crystalline Silicon Interface Passivation Layer by Using Predeposition on the Chamber Wall

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In this study, in situ plasma diagnostic systems of optical emission spectroscopy (OES) and quadrupole mass spectrometry (QMS) were used to monitor an extremely thin (5–10 nm) intrinsic amorphous/crystalline Si interface passivation film as deposited using plasma-enhanced chemical vapor deposition (PECVD). We observed a dramatic improvement in the quality of the passivation layer with a chamber background environment, even with a background pressure of 1E-6 Torr. When the chamber walls were coated (at a predeposition time of approximately 150 min) to a specified thickness of a few hundred micrometers, the minority carrier lifetime increased by more than 26 times, as compared with an insufficiently coated counterpart with a predeposition of approximately 30 min (from approximately 30 μs to 800 μs). In this predeposition process, the tendency of species to concentrate could be systematically obtained using the in situ OES system, with the chamber environment monitored using residual gas analysis and threshold ionization mass spectrometry of QMS. We found that the optimal predeposition time was 150 min, which enabled the OES intensity of SiH4/H2, H2/H2, and H2SiH4 to become stable. OES and QMS were incorporated in this study and were validated using the Fourier-transform infrared spectroscopy absorbance spectra on the films. The plasma condition was stabilized and the minority carrier lifetime was improved to 800 μs. The proposed predeposition process stabilized the chamber environment and gas discharge. Therefore, the optimized value of the minority carrier lifetime could be consistently obtained. A transmission electron microscopy photograph showed a compact a-Si:H layer (of approximately 10 nm) on the c-Si substrate interface passivation layer with a void-free and crystallites-free interface after a predeposition time of 150 min.

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The silicon heterojunction (SHJ) solar cell is one of the most promising solar cell structures, it is highly efficient as well as inexpensive to produce. In particular, the recently reported thin film Si technology enables the development of interdigitated back contact Si heterojunction solar cells with a conversion efficiency exceeding 26.6%. In Si-based solar cell manufacturing, the plasma-enhanced chemical vapor deposition (PECVD) related to the stability of intrinsic hydrogenated amorphous Si (a-Si:H) deposition can be monitored in situ using plasma diagnostics. Furthermore, the insertion of an extremely thin buffer layer (5–10 nm) of a-Si:H as a passivation layer between the wafer and emitter layers (or back surface field (BSF) layer) is the key to a high-performance SHJ solar cell. It is crucial to control the properties of the a-Si:H layers during deposition because the hydrogen atoms in amorphous Si films can be beneficial to filled dangling bonds on a crystalline Si surface, which reduces the defect density and improves the carrier lifetime. Previously, high-quality passivation layer deposition conditions needed to be controlled in the amorphous-crystalline transition zone by optimizing the dilution ratio (H2/SiH4) and pressure. For example, an increase in the dilution ratio over 1 leads to an amorphous-crystalline transition of the film with poor passivation quality. Furthermore, the process window in terms of pressure variation is considerably narrow in that a high-quality film is only achievable within a pressure range of 13.3 Pa centered at 66.7 Pa. Passivation quality is highly sensitive to the reactor state, molecular pump and rotary pump.

Materials and Methods

A parallel-plate radio frequency (RF) PECVD system was used to grow an intrinsic hydrogenated amorphous Si passivation layer (approximately 10 nm) by using a SiH4/H2/Ar gas mixture under 300 m Torr with an RF power of 30W (to avoid damaging the interface from ion bombardment, a low RF power was used) and a hydrogen dilution ratio of [H2]/[SiH4] = 1. Furthermore, the process chamber yielded a base pressure of less than 1E-6 Torr via a coupled turbo molecular pump and rotary pump.
To develop the thin intrinsic amorphous/crystalline Si interface passivation film deposition process, an anticoating device with a honeycomb structure was utilized to ensnarl the built-in OES in the chamber. Additionally, another plasma diagnostic tool of QMS (installed on the other side, as shown in Figure 1) was adopted to quantitatively monitor the plasma conditions to assist the passivation development process.

The intrinsic a-Si:H layers (approximately 10 nm) were deposited on both sides of double side polished 2 × 2 cm² n-type 1–5 Ω cm 300 μm Cz wafers (100) (the lifetime of each sample of the deposited passivation layers was approximately 3.5 μs). Prior to deposition, the wafers were oxidized in an H2O2:H2SO4 (1:2) solution and in 2% hydrofluoric acid for 1 min to remove the oxides. They were then rinsed in deionized water and left to dry in an N2 atmosphere. After the wafer cleaning process, the wafers were immediately transferred into a vacuum chamber of the PECVD apparatus. The effective lifetime was measured using a Sinton Consulting WCT-120 quasi-steady-state photo-conductance lifetime tester in transient mode. The effective lifetime was extracted at an excess carrier density of 10¹⁵ cm⁻³.

Because analyzing the construction of films is challenging, we deposited 180-nm intrinsic a-Si:H layers under the same chamber conditions on one-side polished 2 × 2 cm² n-type 1–5 Ω cm 650 μm Cz wafers (100) to confirm the film’s conditions and plasma interactions using FTIR analysis. A Grant chart of the experimental architecture is shown in Figure 2. TEM micrographs were developed to study the interface. The samples were prepared using a standard ion-milling method.

**OES.**—The emission spectrum of the plasma was detected using OES, and an optical actinometry technique was adopted. The relationship between the intensity of the emission spectroscopy signal \( I_A \) and plasma characteristics is expressed as follows:

\[
I_A \propto n_A \propto [A] \times N_e \propto \int_{E_0}^{\infty} \sigma_A (E) f(e) \, de
\]

The strength of the emission spectroscopy signal \( I_A \) is proportional to the concentration of free radical A \( n_A \), the density of species A \([A]\), the electron density \(N_e\), the cross section of the excited states \(\sigma_A\), and the electron energy distribution function \(f(e)\). Here \(E_0\) is the threshold energy required for excited state \(A^*\).

According to the theoretical derivations, assuming plasma follows a Maxwell–Boltzmann distribution, the intensity of the emission spectroscopy signal can be simplified as a function of the electron energy distribution function (which can be expressed as the electron temperature \(T_e\)), the electron density \(N_e\), and the concentration of measured species \([A]\). The optical actinometry technique was adopted because OES cannot be used to measure the physical characteristics of plasma, such as \(N_e\) and \(T_e\).

Ar (noble gas) was used as a baseline in the OES measurement. The relative concentrations of the measured species can be obtained semiquantitatively by calculating the ratio of the emission spectrum intensity of the excited species to that of the Ar* species (750 nm).

\[
\frac{I_A}{I_{Ar}} = k \frac{[A]}{[Ar]}
\]

Emission lines at 288 (Si), 414 (SiH), 603 (H₂), 656 (H), and 486 nm (H) were mainly used in the current study due to the deposition of a-Si:H layers.

**Fourier-transform infrared spectroscopy.**—An FTIR instrument (Perkin Elmer Spectrum 100) was used to determine the microstructure factor \(R^*\) and hydrogen content \(C_H\). The FTIR absorption spectra were horizontally broken and vertically shifted to highlight the two peaks located at approximately 640 cm⁻¹ and 2100 cm⁻¹, corresponding to the Si-H rocking/wagging and the SiH₆ stretching modes, respectively. The composition of the a-Si:H films, hydrogen content \(C_H\), and microstructure factor \(R^*\) were evaluated using the FTIR results. The \(R^*\) was calculated using the following equation:

\[
R^* = \frac{I_H (2090)}{I_H (2090) + I_H (2000)}
\]

where \(I_H(x)\) is the integrated area of the deconvolved peak centered at \(x\). \(R^*\) is commonly used to characterize the quality of an a-Si:H film, whereby a lower \(R^*\) value indicates that the a-Si:H film has optimal densification.

The total \(C_H\) in the a-Si:H film can be obtained by integrating the area under the rocking mode at 640 cm⁻¹ \(I_{640}\):

\[
C_H = \frac{A_{640} \times I_{640}}{N} = 2.1 \times 10^{19} \text{ cm}^{-2}
\]

where the proportional constant \(A_{640}\) is approximately \(2.1 \times 10^{10} \text{ cm}^{-2}\) and the density of Si atom N = \(5 \times 10^{22} \text{ cm}^{-3}\).

**QMS.**—To quantitatively evaluate the plasma composition, a QMS scan was performed at an ionizer filament current of 20 μA, an electron energy of 70 eV, a mass resolution of 0.1 amu, and a dwell time of 200 ms. The counts were integrated over ±0.5 amu around each mass integer value. Typical residual gas analysis (RGA) spectra in the mass range of 0–100 amu were measured under various chamber conditions (as a function of different deposition times). Furthermore, the threshold ionization mass spectrometry (TIMS) method was applied to estimate the relative concentration of ground-state silane radicals \(\text{SiH}_x, x < 4\) and consumption of \(\text{SiH}_4\).

**Results and Discussion**

**Influence of chamber background conditions.**—According to relevant literature, a good passivation layer process window is very narrow in terms of the hydrogen dilution ratio as well as other processing parameters. The optimization of an extremely thin a-Si:H deposition process is of critical importance because a passivation layer of approximately 10 nm is highly influential to the minority lifetime. After preliminarily adjusting the a-Si:H process parameters, such as the hydrogen dilution ratio, RF power, and process pressure, we found that under the same process parameter settings, the minority carrier lifetime increased by more than 10 times, even as much as 23 times (from 30 μs to 700 μs), which showed that the quality of the thin film was...
unstable (see Figure 3, samples 1–12). Achieving a consistently reliable film with an extremely thin a-Si:H deposition proved challenging. To understand this phenomenon and ensure a stably deposited passivation film, both OES and QMS were used during the extremely thin a-Si:H film deposition process of the intrinsic amorphous/crystalline Si interface passivation.

To ensure that the chamber condition was clean and free from residual film, we used an O2/CF4/Ar plasma cleaning (etching) process to clean the chamber before each experiment. Figure 4 shows the end point detection using an OES intensity of the F∗/O∗ ratio during the O2/CF4/Ar cleaning process. The inset illustrates the spectrum of the chamber condition. From the OES results, intensities of the F∗/O∗ ratio were maintained from 0.9 to 1.4 (average of 1.2) during the end point detection of the etching process. Therefore, the recorded F∗/O∗ ratio was regarded as a standard indicator for thoroughly cleaning the chamber. The baseline for the a-Si:H deposition was proceeded under the processing window of the F∗/O∗ ratio in the range of 0.9 to 1.4.

QMS was adopted to perform a chamber RGA to quantitatively confirm the chamber environment after the cleaning process. Figure 5 shows that the chamber condition was established using the RGAs of QMS, as measured independently over 6 days. The QMS results after the O2/CF4/Ar chamber cleaning process are also shown in the insets. Corresponding to the OES results, QMS further quantitatively demonstrated that the chamber residual gas count commonly existed for atmospheric H2O, N2, O2, and CO2 gases. As shown in Figure 5, the residual gas of the chamber environment was maintained at a specified level after the O2/CF4/Ar chamber cleaning process. Through OES and QMS, these two-step procedures, we established the chamber background conditions for the following that approximately 10 nm passivation layer stability test.

**Process stability of a 10-nm intrinsic passivation layer.**—Based on the established chamber background condition from OES and QMS, we used the same processing parameters (SiH4/H2/Ar gas mixture at 300 m Torr with an RF power of 30W and a hydrogen dilution ratio of [H2]/[SiH4] = 1) to clarify the unstable minority lifetime. As shown in Figure 6, the minority carrier lifetime increased almost linearly as the predeposition time increased, saturating at a predisposition time of approximately 150 min. Therefore, by using this predisposition process, the plasma condition could be stabilized and the minority carrier lifetime could be improved, easily reaching 800 μs. This result can be clarified in future studies by analyzing the deposited thin film conditions (FTIR, microstructure factor R∗, hydrogen content CH%, and TEM) and the correlation with the plasma conditions (OES and QMS).

To confirm the influence of the plasma environment on the film’s property, the film was intentionally grown to 180 nm on one-side polished 2 × 2 cm2 n-type 1–5 Ωcm 650 μm Cz wafers (100) in the same chamber state for IR analysis (because of a limited bench analysis capability). Figure 7 shows the FTIR absorption spectra of the a-Si:H films deposited as a function of the predeposition time. The signals located at approximately 640 cm−1 and 2000 cm−1 were assigned as the wagging/rocking and stretching modes of the Si-H bond, respectively. The signal located at approximately 1100 cm−1 was assigned as the Si-O-Si bond.26 The absorption peaks of the deposited films from 1900 cm−1 to 2200 cm−1 were used to extract the different modes, such as Si-H

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**Figure 2.** Gantt chart of the experimental sampling time architecture.

**Figure 3.** Unstable phenomenon on the minority lifetime of an extremely thin a-Si:H passivation layer with the same processing parameters.

**Figure 4.** Establishment of chamber background conditions: end point detection from an optical emission spectrometry intensity of the F*/O* ratio during the O2/CF4/Ar chamber cleaning process, as shown in the inset. Intensities of the F*/O* ratio were maintained from 0.9 to 1.4 (average of 1.2) during the end point detection of the etching process.

**Figure 5.** End point detection by OES (- clean process)
Figure 5. Chamber background condition as established by the residual gas analysis from quadrupole mass spectrometry. The quadrupole mass spectrometry results after the O₂/CF₄/Ar chamber cleaning process are shown in the inset.

Figure 6. Effect of the predeposition time on the minority lifetime of the a-Si:H passivation layer.

Figure 7. Fourier-transform infrared spectroscopy spectra of the a-Si:H thin films deposited on the c-Si substrates as a function of the predeposition time.

Figure 8. Microstructure factor (R*) and hydrogen content (%CH) in a-Si:H films as a function of the predeposition time. The inset shows the deconvolution procedure for calculating R*.

Figure 9. Cross-sectional TEM micrograph of the a-Si:H/c-Si interface at a predeposition time of 150 min. As shown, a defect-free, epitaxial-absent layer was found between the a-Si:H/c-Si interface, which enabled a smooth and discernable a-Si:H/c-Si heterojunction to develop. Moreover, this impact was correlated with a low intensity of Si-H₂ bonds, which enabled a compact microstructure that was uniformly distributed with a clear interface free from voids and crystallites in the a-Si:H film.

Plasma diagnostic of a 10-nm intrinsic passivation layer process.—Because of the different film qualities under the same process parameters, we investigated the relationship between the passivation and plasma characteristics. As presented in Figure 10a, when the predeposition time increased from 5 min to 30 min, the intensity ratio of Hα/Ar* and Hβ/Ar* decreased. By increasing the predeposition time to over 30 min, the Hα/Ar* and Hβ/Ar* became saturated. This indicated that the source of the H atoms in the plasma was reduced as a function of the predeposition time. The intensity ratio

mono-hydrides (approximately 2000 cm⁻¹) and Si-H₂ clusters (approximately 2100 cm⁻¹). Furthermore, the quality of the film’s microstructure was routinely used as the indicator of the microstructure factor (R*).

As shown in Figure 7, when the predeposition time increased incrementally, the signals located at approximately 640 cm⁻¹ and 1100 cm⁻¹, the peaks of the FTIR absorption spectra decreased slightly, whereas for the peak of the Si-H bond (stretching mode) at 2000 cm⁻¹, the highest peak was located at a predeposition time of 150 min. For the peak at 1100 cm⁻¹, a predeposition time of 30 min produced the highest peak. This phenomenon may be attributed to the residual water present in the chamber, which was elucidated in the QMS analysis. Moreover, for the peak at approximately 2000 cm⁻¹, the highest peak was located at a predeposition time of 150 min, which was correlated with the microstructure factor R*.

The corresponding microstructure factor R* and hydrogen content C_H% values are presented in Figure 8. As shown, the hydrogen content C_H% values were maintained at approximately 10.5 and the lowest R* value (0.02) was obtained at a predeposition time of 150 min. This means that the Si/H composition ratio of the film did not change; only the film’s structure changed from Si-H₂ to Si-H. In the passivation of the extra thin film, a low R* value was favorable because H atoms mainly existed in the Si-H form of the a-Si:H matrix. We performed cross-sectional TEM to investigate the properties of the interface to confirm the quality of the interface between the Si wafer and passivation layer. Figure 9 shows a cross-sectional TEM micrograph of the a-Si:H/c-Si interface at a predeposition time of 150 min. As shown, a defect-free, epitaxial-absent layer was found between the a-Si:H/c-Si interface, which enabled a smooth and discernable a-Si:H/c-Si heterojunction to develop. Moreover, this impact was correlated with a low intensity of Si-H₂ bonds, which enabled a compact microstructure that was uniformly distributed with a clear interface free from voids and crystallites in the a-Si:H film.
of SiH<sup>∗</sup>/Ar<sup>∗</sup> and Si<sup>∗</sup>/Ar<sup>∗</sup> increased proportionally with the predeposition time from 5 min to 60 min. By increasing the predeposition time from 90 to 150 min, the intensity ratio of SiH<sup>∗</sup>/Ar<sup>∗</sup> and Si<sup>∗</sup>/Ar<sup>∗</sup> became saturated. Under fixed process conditions, these phenomena were potentially caused by residual moisture (H<sub>2</sub>O) released from the chamber wall. The residual moisture on the chamber absorbed part of the energy and caused a low dissociation of SiH<sub>4</sub>. As the predeposition time increased, the chamber wall became covered in a thin film, moisture was consumed, and the energy originally consumed by the moisture shifted to SiH<sub>4</sub>, gradually increasing SiH<sup>∗</sup>/Ar<sup>∗</sup> and Si<sup>∗</sup>/Ar<sup>∗</sup>. This indicated that more silane was decomposed into plasma and then a high deposition rate deduced could be obtained; furthermore, the plasma was in a relatively high depletion status than 0 to 30 min but can be experimentally correlated with the silane consumption as measured by QMS.

The Si<sup>∗</sup>/SiH<sup>∗</sup>, H<sub>2</sub>/SiH<sup>∗</sup>, and H<sub>β</sub>/H<sub>α</sub> curves, as a function of the predeposition time, are shown in Figure 10b. Under the fixed process parameters, the intensity ratio of H<sub>2</sub>/SiH<sup>∗</sup> and Si<sup>∗</sup>/SiH<sup>∗</sup> decreased abruptly when the predeposition time increased from 5 min to 30 min, until saturation occurred (after a predeposition time of 90 min). The ratio of H<sub>2</sub>/H<sub>α</sub> increased slightly before 30 min, which indicated that the proportion of dissociated HX-related species remained almost constant. The ratios of silane to the provided H<sub>2</sub> and H<sub>α</sub> were fixed.

However, the change from H<sub>2</sub>/SiH<sup>∗</sup> indicated that not only the process parameters but also another source provided H in the chamber. Because Si<sup>∗</sup>/SiH<sup>∗</sup> can be considered an electronic temperature trend indicator, the result suggested that the reduced ion bombardment potentially reduced the epitaxial layers. This might have been related to the consumption of the residual water in the chamber, which was supported by the FTIR and QMS analyses.

We investigated the relationship between the passivation and plasma characteristics. Figure 11a presents the time series of the RGA data as a function of the predeposition time obtained from the QMS results, which revealed that the higher silane related reactive species (HSRS) were comparatively depleted in the PECVD process. The absence of HSRS in this plasma state occurred because the Si-H<sub>2</sub> structure and void generation rate were reduced considerably. Figure 11b shows the time-dependent intensities of H<sub>α</sub>, H<sub>β</sub>, and H<sub>2</sub>O, which illustrate the reduced H<sub>2</sub>O intensity (18 amu), and simultaneously increased hydrogen H<sub>α</sub> and X < 2 (amu) intensities. This trend confirmed that the residual water in the chamber could be minimized by increasing the predeposition time. The obtained relative density trends of SiH<sub>x</sub> (x < 4) radicals under different predeposition times from the TIMS measurement are shown in Figure 12. When the predeposition time increased from 30 min to 150 min, the relative density of the SiH<sub>x</sub> (x < 4) radicals became saturated. This impact corresponded with the results obtained from OES and QMS, which showed that the water residue could be minimized by increasing the predeposition time.

Finally, as presented in Figure 13, increasing the predeposition time (from 30 min to 90 min) promoted the dissociation of silane with a high deposition rate. When the predeposition time was continuously increased to 150 min, the deposition rate started to saturate, which suggested that the plasma was in a high depletion status.

In addition, the extremely thin a-Si:H passivation films (approximately 10 nm) deposited under the controlled plasma conditions exhibited were of high quality, as shown in Figure 9. This finding is consistent with another study, which showed that controlling plasma processing parameters to just below the onset of the amorphous-to-crystall transition achieves a passivation with a relatively high depletion fraction of SiH<sub>3</sub>. Furthermore, a positive correlation between SiH<sub>2</sub> consumption and the deposition rate was obtained because SiH<sub>3</sub> is a long-lifetime radical and a main precursor in thin film deposition, which is in an agreement with our results.

Conclusions

Continuously performing thin film deposition experiments under the same process parameters inevitably changed the chamber environment and plasma conditions, even with a background pressure of 1E-6 Torr. Furthermore, the chamber surface enclosing the plasma was synergistically influential on the plasma’s chemistry interactions between molecules and atoms from the reactive surfaces and chamber walls, as well as the surface etching of the ion and radical associations at the surface, are all part of the complicated plasma chemistry in the PECVD process. The combined impact can be incorporated into
the residual water molecules with related dissociation molecules and ions. Therefore, the plasma diagnostic tools of OES and QMS were incorporated in this study to investigate the effect of the predeposition time on the quality of the thin film. The results showed that in the first 30 min of predeposition, water molecules and ions became precursor radicals in the plasma region, resulting in a higher Si-O-Si bond in the film, which was confirmed by the FTIR absorbance spectra.

According to the measurement results of SiH/ SiH+ from OES, a relatively high electronic temperature caused a relatively high probability of ion bombardment and a hydrogen etching effect. Thus, a relatively low reaction and deposition rate were obtained. When the predeposition time increased from 30 min to 150 min, the relative density of SiHx (x < 4) radicals decreased dramatically, which became saturated until a deposition time of 150 min. Minimizing residual water by increasing the predeposition time is the main contributing factor, which was confirmed from the OES and QMS measurements. Overall, the optimized PECVD processing conditions with an improved charge carrier lifetime were iteratively obtained at the a-Si:H(i)/c-Si-interface by the chemical passivation of saturated dangling bonds. By prudently using the proposed predeposition process, the plasma condition can be stabilized and the minority carrier lifetime can be improved easily to 800 μs. Efficient plasma diagnostic tools are of great value because in-situ process monitoring of the passivated surfaces prone to epitaxial growth and voids can be prevented, as confirmed from the TEM results.

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