Chemical-reaction dependence of plasma parameter in reactive silane plasma

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Abstract

Electron temperature in a silane glow-discharge plasma, being an important plasma parameter for determining photo-induced instability in the resulting hydrogenated amorphous silicon (a-Si:H), has been studied under various film-preparation conditions. We have used an optical-emission-intensity ratio of Si I to SiH²⁺ (I₆/I₁₃) which corresponds to the high-energy-tail slope of the electron-energy-distribution function in the plasma as a measure of electron temperature in a reactive silane glow-discharge plasma. We have found quite differently from the conventional non-reactive glow-discharge plasma such as hydrogen plasma that the electron temperature in the silane plasma is strongly modified by the substrate temperature (gas temperature) especially under high silane-gas partial-pressure condition. This anomalous behavior of the electron temperature in the silane plasma has been explained by means of gas-phase-polymerization reaction and electron-attachment process to the polymers in the plasma. The electron temperature has been remarkably reduced when a hydrogen-dilution method and a cathode-heating method are used which are considered to control polymer-formation reactions in the silane plasma together with utilization of conventional electron-temperature-controlling methods such as a very high plasma-excitation frequency and an application of magnetic field for electron-confinement. As a consequence of the reduction of electron temperature in the silane plasma, highly stabilized a-Si:H has been successfully obtained even under high growth rate conditions of 1.5 nm s⁻¹. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Reactive silane plasma; Higher silane related reactive species; Hydrogenated amorphous silicon; Photo-induced instability; Electron temperature; Polymerization reaction; Electron attachment

1. Introduction

Hydrogenated amorphous silicon films (a-Si:H) have been widely used for thin film silicon based semiconductor devices such as solar cells and thin film transistors. High growth rate of a-Si:H more than 1 nm s⁻¹ is thought to be crucial for reducing production cost of these devices. However opto-electronic properties, for instance, energy-conversion efficiency of a-Si:H based solar cells is severely deteriorated by prolonged light soaking especially when the a-Si:H is prepared at high growth rate [1]. Although possible origins for a severe photo-induced degradation in a-Si:H have been proposed so far [2,3], definitive conclusion has not yet been given. Recently we have found that there is an intimate relationship between degree of photo-induced degradation and density of Si–H₂ bonding configuration incorporated in the film among a variety of a-Si:H prepared under various deposition conditions especially at high growth rate of 1.5–2.0 nm s⁻¹, as shown in Fig. 1 [4]. We have also found that the contribution of higher silane related reactive species (HSRS) such as Si₃H₈ during film growth leads to an excess incorporation of Si–H₂ bonding configuration in the resulting a-Si:H [1,5]. We have also predicted through a theoretical consideration using a couple of reaction rate equations that the contribution ratio of HSRS during film growth is strongly affected by the electron temperature in the silane plasma [6]. Namely, electron temperature in the silane plasma is a key plasma parameter for obtaining highly stabilized a-Si:H [6,7].

In this paper, we show our recent understandings of a-Si:H-film-growth process from silane glow-discharge plasma and the relationship between plasma parameter and degree of photo-induced degradation in the resulting films. Finally we demonstrate the successful control of silane reactive plasmas to prepare highly stabilized a-Si:H using electron-temperature-controlling methods through the control of polymerization reactions in the plasma together with conventional electron-temperature-controlling methods.
showing a steady-state density of around 10^{12} \text{cm}^{-3} [8].
SiH_2 radicals react with silane molecules forming di-silane,
this di-silane reacts with another SiH_2 radical forming tri-
silane as is seen in the sketch in Fig. 3. In this way, higher
silane molecules including many Si atoms, (hereafter, in this
paper higher silane molecules such as Si_3H_6, Si_4H_{10}, Si_5H_{12}
are denoted as silane polymers to distinguish from reactive
HSRS such as Si_4H_9) are formed through successive inser-
tion reactions of SiH_2 radicals. As has been observed experi-
mentally [9], particle formation more than 10 nm in size is,
therefore, observed near the SiH_2-radical-generation space.
The HSRS such as Si_3H_9 is formed by a dissociative-
electron impact to silane polymers such as Si_4H_{10}.

3. Electron temperature measurement in silane plasmas

Assuming that Si_4H_9 is the responsible HSRS for producing
a specific network structure (excess Si–H bonding configu-
ration in the resulting film) during film growth, the contribution
ratio of HSRS to film growth is expressed as Eq. (1) which is
estimated by a couple of reaction-rate equations [6],

\[
\frac{[\text{Number density of HSRS}]}{[\text{Number density of SiH}_3]} = \frac{[\text{Si}_4\text{H}_9]}{[\text{SiH}_3]} = k_2 k_3 k_4 \tau_6 \tau_7 \tau_{10} N_e \sigma_v v_e [\text{SiH}_2]^3 \\
\times [\text{SiH}_3] \\
\propto [\text{SiH}_2]^3 [\text{SiH}_4]/[\text{SiH}_3] \\
= \frac{(N_e \sigma_v v_e [\text{SiH}_4])^3}{((R + 1)k_2 [\text{SiH}_4])^3 N_e \sigma_v v_e \tau_3} \propto \frac{N_e^3}{N_e (R + 1)^3} 
\]

where \( N_e \) and \( N_r \) are the electron densities responsible for the

![Fig. 1. Relationship between degree of photo-induced degradation and hydrogen content of Si–H bonding in the resulting a-Si:H prepared by various deposition conditions. The films are evaluated from the photo I–V characteristics of Ni–a-Si:H Schottky diode.](image)

![Fig. 2. Primary electron-induced dissociative-excitation-reaction process of SiH_4 molecules in the reactive silane plasmas. All the species are produced from each electronic excited state spontaneously via one-electron-impact dissociation passway.](image)
dissociative excitation of SiH₂ molecule into SiH₃ (electron energy higher than 9.47 eV) and SiH₄ into SiH₃ (higher than 8.75 eV) [10], R is hydrogen-dilution ratio which is defined as \( R = \frac{k_1[H_2]}{k_2[SiH_4]} \), \( k_1 \), \( k_2 \), \( k_3 \) and \( k_4 \) are the reaction-rate constants of SiH₂ with H₂, SiH₃ with SiH₄, SiH₂ with Si₃H₈, and SiH₂ with Si₃H₈, respectively, \( \tau_3 \), \( \tau_6 \), \( \tau_9 \), \( \tau_{10} \) are the characteristic diffusion lifetimes of SiH₁, Si₃H₈, Si₃H₈, Si₃H₈ and Si₃H₁₀, respectively. \( N_e \) is the electron density for the decomposition of Si₃H₁₀, \( \sigma_2 \), \( \sigma_3 \) and \( \sigma_9 \) are the dissociation-cross section of SiH₂, SiH₃ and Si₃H₁₀ and \( v_e \) is the thermal velocity of electrons. Since electron-energy-distribution function (EEDF) in plasma is thought to obey Maxwellian, \( N_e/N_e^* \) gives the slope of high-energy tail of EEDF, i.e. a higher \( N_e/N_e^* \) gives a less steeper slope. Consequently, \( N_e/N_e^* \) is a proportional function of electron temperature in the silane plasma. Therefore, the contribution ratio of HSRS to the film growth is strongly dependent on the electron temperature in the silane plasmas.

To measure the electron temperature in the silane plasma, we use an optical-emission-intensity ratio of \( I_{[Si]}/I_{[SiH]} \) to
avoid an experimental ambiguity in using Langmuir probe method due to a semiconductor-film deposition on the probe. The optical emission intensity ratio $I_{Si}/I_{SiH}$ is also expressed by

$$\frac{[Si^{+} \text{emission intensity}]}{[SiH^{+} \text{emission intensity}]} = N_{e_A} \sigma_{A} v_{e} [SiH_{4}] / N_{e_B} \sigma_{B} v_{e} [SiH_{4}] \propto N_{e_A} / N_{e_B}$$

(2)

where $N_{e_A}$ and $N_{e_B}$ are the electron densities responsible for the dissociative excitations of SiH$_4$ molecules forming Si$^{+}$ and SiH$^{+}$, $\sigma_{A}$ and $\sigma_{B}$ are the dissociative-excitation-cross sections for Si$^{+}$ and SiH$^{+}$, respectively. The emissive species of Si$^{+}$ and SiH$^{+}$ are produced also via one-electron-impact dissociation process of SiH$_4$ molecules with electron energies of more than 10.53–11.5 and 10.33–10.5 eV, respectively [10,11], as is shown in Fig. 1, and an electron-energy dependence of Si$^{+}$ and SiH$^{+}$ emission-cross sections exhibits a similar trend [11]. Therefore, the optical-emission intensity ratio of $I_{Si}$ to $I_{SiH}$ gives also an information about the slope of high-energy tail of EEDF in the plasma. The order of electron energies for the formation of SiH$_3$, SiH$_2$, SiH$^{+}$ and Si$^{+}$ are described in Fig. 4. Namely, when the $I_{Si}/I_{SiH}$ takes larger value, the slope of high-energy tail of EEDF should be less steep (higher electron temperature).

To confirm the experimental applicability of the optical-emission-measurement method, electron temperature in hydrogen plasma was measured using both Langmuir-probe method and optical-emission method ($I_{FIR}/I_{FIR}$ in this case). As a result, same trends of electron temperature as a function of plasma excitation frequency and pressure were obtained.

4. Anomalous behavior of electron temperature in silane plasmas

Using a conventional capacitively coupled plasma-enhanced chemical-vapor deposition reactor equipped with parallel plate electrodes, power density, plasma excitation frequency, gas flow rate, gas pressure, substrate temperature, and electrode distance are listed up as controllable deposition conditions. Among them, very high plasma-excitation frequency (VHF) and higher gas pressure are known as effective deposition conditions to reduce the electron temperature in general. Fig. 5(a) shows the electron temperature measured by the optical-emission-intensity ratio of $I_{Si}/I_{SiH}$ plotted against plasma-excitation frequency in the silane plasma. In this measurement we used high growth-rate conditions (∼1.5 nm s$^{-1}$), i.e. a silane-gas-flow rate of 30 sccm, an electrode distance of 20 mm, a silane-gas pressure of 50 mTorr, and a substrate temperature ($T_s$) of 250°C with a changing power density applied to the plasma to keep the growth rate constant. The experimental setup used in this study has been shown elsewhere [12]. Electron temperature measured by $I_{Si}/I_{SiH}$ in the silane plasma is drastically decreased with increasing plasma-excitation frequency due to low loss probability of ions, which leads to low loss rate of electrons. Remarkable improvement of photo-induced degradation in the resulting a-Si:H prepared under the same deposition conditions as the electron temperature measurement with increasing plasma-excitation frequency is seen in Fig. 5(b). In this case a Fill Factor (FF) after light soaking in Schottky diode (Ni(5 nm)/a-Si:H(1 μm)) is used as the measure of stability of a-Si:H against light soaking, whose details have also been reported in Refs. [13,14]. It is concluded from the results shown in Fig. 5 that VHF plasma-excitation frequency is one of the useful deposition conditions for preparing stable a-Si:H through the reduction of electron temperature in the silane plasma.

It is expected that gas pressure is another useful deposition condition to reduce the electron temperature through the reduction of electron loss rate, leading to high stability in the resulting a-Si:H. Fig. 6 shows the relationship between electron temperature measured by the optical-emission-intensity ratio and the gas pressure for different substrate temperatures for: (a) silane plasma and (b) hydrogen plasma. For a conventional non-reactive plasma such as hydrogen plasma, electron temperature should be reduced with increasing gas pressure due to low ambipolar-diffusion
loss rate of electrons in the plasma. As in the same sense, when decreasing the substrate temperature, electron temperature is expected to be reduced due to an increase of effective gas density. The expected trends against gas pressure and substrate temperature are clearly seen in Fig. 6(b). However, the behavior of electron temperature in silane plasma is quite anomalous in contrast to that in hydrogen plasma. The electron temperature in the silane plasma increases with increasing silane gas pressure especially in higher pressure range, moreover lower electron temperature at higher substrate temperature is remarkably seen under higher pressure conditions. The change in the electron temperature with changing pressure and substrate temperature in the silane plasma is not easily understood, although there are some papers proposing the increase of electron temperature due to the presence of an electron-attachment process to large particles more than 6 nm in diameter [15].

5. Possible model for explaining anomalous behaviors of electron temperature

To investigate the cause of anomalous behaviors of electron temperature against gas pressure and substrate temperature, time evolution measurement of electron temperature in the silane plasma was carried out for different substrate temperatures under the same silane gas pressure condition of 100 mTorr. Fig. 7(a) depicts the time evolutions of the electron temperature measured by $I_{e}/I_{e+}$ after turning on the silane plasma. The case of hydrogen plasma is also shown in Fig. 7(b) as a reference. The electron temperature in the silane plasma at $T_s = 150^\circ$C starts to increase gradually after turning on the plasma, followed by a saturation, while it keeps a constant value for the case of $T_s = 400^\circ$C. In contrast, the electron temperature in the hydrogen plasma shows a constant value for all the substrate temperatures from the start in the present experimental time resolution.

A tentative model for the increase of electron temperature with decreasing substrate temperature and increasing gas pressure is proposed [16] as sketched in Fig. 8. Formation reaction process of silane polymers such as di-silane, tri-silane, and tetra-silane consists of successive insertion reactions of SiH$_2$ reactive radicals into silane, di-silane, and tri-silane through a three-body reaction in the silane plasma. When the silane polymer reaches a critical size, low energy electrons attach these polymers since such a large size polymer is recognized as a solid in the plasma, which leads to an increase of electron loss rate in the plasma. As a consequence, electron temperature increases

Fig. 6. Electron temperature measured by optical-emission-intensity ratio as a function of gas pressure in: (a) silane and (b) hydrogen plasmas for different substrate temperatures of 150, 250, and 400°C.

Fig. 7. Time evolution of the electron temperature measured by optical-emission-intensity ratio in: (a) silane and (b) hydrogen plasmas at different substrate temperatures of 150, 250, and 400°C.
due to high electric-field application to the plasma caused by an increase of the plasma impedance through the reduction of electron density. When the electron temperature increases once, SiH₂ radical production is promoted in the plasma because it needs higher electron energy as compared to film precursor SiH₃ as shown in Fig. 4, then the formation rate of silane polymers is enhanced. When the density of silane polymers is increased, electron-attachment rate is enhanced, leading to an increase of electron temperature through an increase of electron-loss rate. Thus, the increase of electron temperature in the silane plasma is continued by this feed-back phenomenon.

Since the insertion reactions of SiH₂ with silane, di-silane, and tri-silane exhibit negative activation energy due to their three-body-reaction characteristics [17], formation-reaction rate of silane polymers is strongly influenced by the temperature of third bodies (other ambient SiH₃ molecules in this case). Using the model mentioned above, anomalous behaviors of electron temperature against substrate temperature and gas pressure shown in Fig. 6(a) are successfully explained. Namely, formation rate of silane polymers is reduced at higher substrate (gas) temperature, leading to a realization of low electron temperature plasma through a suppression of the feed-back phenomenon shown in Fig. 8.

6. Responsible silane polymers to increase the electron temperature

To clarify the critical size of silane polymers that contributes to the electron-temperature rise through the electron attachment process, mass spectrometry was used whose details is also seen in Ref. [18]. Time evolution of mass–signal intensities of \([m/e] = 30, 60, 90, 120,\) and 150 after turning on the plasma was taken in the same time scale as that of electron temperature time evolution.

Fig. 9(a) and (b) show the time evolution of \([m/e] = 30, 60, 90, 120,\) and 150 corresponding to the number densities of silane, di-silane, tri-silane, tetra-silane, and penta-silane, respectively [18], after tuning on the silane plasma for different substrate temperatures of: (a) 150 and (b) 400°C.
The number densities of silane polymers are calculated using the ratio of each signal intensity to the signal intensity of [m/e] = 30. Signal intensity of [m/e] = 30, corresponding to the number density of silane molecules in the plasma, decreases slightly with time due to a consumption of silane molecules in the plasma. Signal intensity of [m/e] = 60 increases with time by the formation of di-silane molecules in the plasma. However, it should be noted here that di-silane molecule exists in the silane gas cylinder from the start, therefore this slope does not show the formation rate of di-silane molecule directly. Signal intensities of [m/e] = 90, 120 and 150 increase with time at \( T_s = 150^\circ C \) as shown in Fig. 9(a) demonstrating the formation of silane polymers in the plasma, while signal of [m/e] = 150 at \( T_s = 400^\circ C \) does not appear at all as is seen in Fig. 9(b). Thinking of the absolute number density of silane polymers to affect the electron temperature, electron-attachment-loss rate should be almost of the same order as the electron-ambipolar diffusion-loss rate. The electron-ambipolar diffusion-loss rate and the electron-attachment-loss rate are expressed by Eqs. (3) and (4).

Electron-ambipolar diffusion-loss rate: \( N_e/\tau = N_e D_{amb}/l^2 \)  

Electron-attachment-loss rate: \( \sigma_{att} v_e N_e [Si_nH_m] \)  

where \( N_e \) is the electron density, \( \tau \) the characteristic lifetime of electrons, \( D_{amb} \), the ambipolar diffusion coefficient which is limited by a slow diffusion of \( SiH_n^+ \) ions, \( l \) the characteristic diffusion length, \( \sigma_{att} \) the electron-attachment-cross section to silane polymers, \( v_e \) the thermal velocity of electrons, and \( [Si_nH_m] \) is the number density of silane polymers, respectively. When using typical values of \( D_{amb} = 100 \text{ cm}^2 \text{ s}^{-1} \), \( l = 4 \text{ cm} \), \( \sigma_{att} = 10^{-16} \text{ cm}^2 \), and \( v_e = 10^5 \text{ cm s}^{-1} \), the critical number density of silane polymers to cause the electron-temperature rise through the electron-attachment event is calculated to be \( 6.0 \times 10^8 \text{ cm}^{-3} \). As compared to Fig. 9(a) and (b), the number density of \( Si_3H_{12} \) (m/e = 150) for \( T_s = 150^\circ C \) and that of \( Si_5H_{10} \) (m/e = 120) for \( T_s = 400^\circ C \) reach the critical number density calculated here at 0.5 s after tuning on the plasma. Thus these silane polymers (\( Si_3H_{12} \) and \( Si_5H_{10} \)) are considered to cause the electron temperature rise. However, the increase of electron temperature is seen only at substrate temperature of \( 150^\circ C \) from 0.5 s after tuning on the plasma as shown in Fig. 7(a). Consequently, it is suggested from the results that the responsible silane polymers produced in the silane plasmas for affecting the electron temperature through the electron-attachment events is penta-silane (\( Si_5H_{10} \)) molecule. Therefore, it is indicated that the reduction of electron temperature in the silane plasma which is the key parameter to improve the stability of resulting a-Si:H should be carried out not only by using conventional electron-temperature-controlling methods such as plasma-excitation frequency and gas-pressure but also by using a specific method to control penta-silane-formation reactions in the plasma by some means such as gas heating, hydrogen dilution, etc.

7. Effect of hydrogen dilution on electron temperature

Hydrogen-dilution method (silane source gas is diluted with hydrogen) has been widely used for improvement of photo-induced degradation properties of resulting a-Si:H [5,19,20]. Although the role of hydrogen dilution is still controversial, we can propose a possible role of hydrogen dilution using the model mentioned above to explain the anomalous electron temperature behavior in the silane plasma. Hydrogen molecules react with \( SiH_2 \) radicals, which are the triggering radical species for the polymerization reaction in the plasma, although the reaction rate constant of \( SiH_2 + H_2 \rightarrow SiH_4 \) is two orders of magnitude smaller than that of \( SiH_2 + SiH_4 \rightarrow Si_2H_6 \) [21,22]. The reaction rate for the reaction of \( SiH_2 + H_2 \rightarrow SiH_4 \) becomes faster than that for the formation reaction of silane polymers such as \( SiH_2 + Si_3H_8 \rightarrow Si_5H_{10} \), because the number density of \( H_2 \) molecules is much higher than that of \( Si_3H_8 \) and \( Si_5H_{10} \) molecules as shown in Fig. 9. Therefore, the increase of electron temperature through the electron-attachment process to penta-silane polymerized molecule is suppressed by the presence of recombination reaction of \( SiH_2 \) with hydrogen.

Fig. 10 shows the experimental evidence of the effectiveness of hydrogen-dilution method. Reduction of electron temperature against hydrogen dilution ratio is obviously seen in the figure and we have also demonstrated the improvement of stability of resulting a-Si:H [6]. The increase of electron temperature with further increase of \( H_2 \)-dilution ratio as is also seen in the figure is explained by the change of dominant ionic species from \( SiH_2^+ \) to \( H_2^+ \). Namely, the electron temperature is increased by an increase of electron-diffusion-loss rate due to larger ambipolar diffusion coefficient of hydrogen ions as compared to that of \( SiH_2^+ \) ions toward higher \( H_2 \)-dilution range.

Fig. 10. Electron temperature measured by optical emission-intensity ratio as a function of hydrogen-dilution ratio.
8. Relationship between electron temperature in silane plasma and photo-induced degradation in resulting a-Si:H

It is expected that gas heating is another suitable method to decrease the electron temperature, since the formation reactions of silane polymers show negative temperature dependence due to their three-body reactions. In order to confirm the effect of gas heating on the electron temperature in silane plasmas, cathode-heating method has been used, where the cathode is heated up to 230°C while keeping the substrate temperature the same at 250 or 300°C. The results are summarized in Fig. 11 together with results of several electron-temperature-controlling methods such as utilization of VHF as the plasma-excitation frequency, high total pressure, and magnetic field for electron confinement [23]. Fig. 11 shows the relationship between the electron temperature in silane plasmas and the stability of resulting a-Si:H (the difference in the fill factor of photo-I–V characteristics of Schottky diodes between before and after light soaking, showing the degree of photo-induced degradation) for a variety of silane plasmas mentioned above. As is seen in the figure, the stability of resulting a-Si:H is drastically improved by the reduction of electron temperature in the silane plasma by many means during film growth.

9. Conclusions

The key issue to be solved for obtaining highly stabilized amorphous silicon at high growth rate is discussed in this paper. We have arrived at the following observations: (1) there exists almost one-to-one correspondence between photo-induced instability and Si–H₂ density incorporated in a-Si:H. (2) The Si–H₂ density in a-Si:H is strongly affected by the contribution ratio of HRS such as Si₃H₆ during film growth while keeping the substrate temperature constant. (3) The contribution ratio of HRS during film growth is influenced by electron temperature in the silane plasma, which is predicted using a couple of rate equations. Therefore, it is concluded that one of the most important key issue to obtain highly stabilized a-Si:H is to reduce the electron temperature in the silane plasma during film growth.

However, we have also found that the electron temperature measured by optical-emission-intensity ratio (Iₛ/IₛH) exhibits anomalous behavior against substrate (gas) temperature and silane-gas pressure. This anomalous behavior has been explained by taking into account silane-polymer formation and electron attachment events to these polymers. Using time evolution experiments in the mass spectrometry, responsible size of silane polymers, which is recognized as solids in the plasma, has been assigned to be penta-silane. To suppress the polymerization reaction in the silane plasma, hydrogen-dilution and cathode-heating methods have been used. Together with other conventional electron-temperature-controlling methods, we have successfully obtained highly stabilized a-Si:H at growth rates as high as 1.5 nm s⁻¹.

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