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Electron-impact silane dissociation and deposition rate relationship in the PECVD of microcrystalline silicon thin films

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Abstract: An investigation of the relation between silane electron impact dissociation and deposition rates of microcrystalline silicon thin films, has been performed in highly diluted SiH_4 in H_2 discharges, by applying a combination of experimental measurements and modeling of the process. A wide range of frequencies (13.56 MHz – 50 MHz), power densities (11 mW/cm^2 – 162 mW/cm^2) and silane partial pressures (2% - 6%) at two total SiH_4/H_2 pressures of 0.5 Torr and 1 Torr has been studied. In the lower pressure, independent of all other discharge parameters, SiH_4 primary dissociation has been found to be responsible for about 70% of the total silane consumption in the discharge, while a fraction of 12% of the initially produced silicon hydrides are incorporated into the growing film. The increase of pressure leads to a drop of the contribution of the SiH_4 primary dissociation to the total silane consumption and to an increase of the deposition efficiency of the initially produced radicals to a value of 26%. This result is attributed to the production of additional, two silicon atom precursors via secondary gas-phase reactions.

1. INTRODUCTION

Microcrystalline silicon thin films (μc-Si:H) deposited from highly diluted SiH_4 in H_2 discharges have very recently found applications in solar cells [1] and thin film transistors [2]. A key point towards the control of this process lies in understanding the basic phenomena that take place in the gas phase. More precisely, the effect of various plasma parameters (power, pressure, frequency, gas mixture) on the dissociation of silane towards neutral and charged species is of primary importance, because the degree of dissociation and the kind of the resulting species will largely determine the film growth rate and properties. However, the effect of these parameters on SiH_4 dissociation and the relation between SiH_4 dissociation and the film growth rate is not completely clear despite the large research effort spend on the very similar amorphous silicon (a-Si:H) deposition process during the past years. Indeed, the existing literature concerning the issue includes simulations of SiH_4 and SiH_4/H_2 discharge kinetics [3-6] and experimental measurements of the total SiH_4 dissociation under a variety of discharge conditions [7-11].

The above-mentioned theoretical studies are mainly focused on the development of self-consistent process models including gas-phase physics and gas-phase and surface chemistry. Although these models have helped a great deal in understanding the physical and chemical mechanisms that lead to SiH_4 dissociation and film growth they have not been able to provide a relation between the film growth rate and SiH_4 dissociation rate, due to the quite large number of approximations required. In addition, the lack of comparison or the disagreements between model and experimental results [3-6] have prevented the extraction of unambiguous conclusions concerning the issue.

On the other hand, total SiH_4 dissociation in SiH_4 and SiH_4/H_2 discharges has been previously measured, using mass spectrometry [8-11] and Fourier transform infrared (FTIR) absorption spectroscopy [7]. However, since SiH_4 consumption in these discharges is not only due to electron impact dissociation/ionization but it is also possible through secondary reactions with H atoms and other radicals and ions, further analysis of the gas phase chemistry was required for the interpretation of these measurements. In two of the cited cases [9,10] the results were accompanied by a qualitative discussion of the basic dissociation channels, while in the rest of the cases [7,8,11] simplified models, neglecting ion chemistry and/or radical-SiH_4 reactions, were applied for the distinction of the different consumption paths. According to these, the increase of power [8], the increase of frequency [7,11] and the decrease of the total gas pressure [8,10] have been found to enhance SiH_4 dissociation rate. Nevertheless, with only one exception [7], no attempt was made to correlate SiH_4 dissociation rate with the film growth rate.

The main purpose of the present work is to investigate the relation between gas phase processes leading to SiH_4 dissociation and the output of the process. This purpose is accomplished by applying several experimental techniques combined with a detailed simulation of the gas phase and surface chemistry in highly diluted SiH_4 in H_2 discharges. Special attention has been given to avoid experimental uncertainties which can appear in the measurement of the effective power consumed in the discharge [12], by using an accurate power and impedance determination method [13]. In addition, mass spectrometry has been used for the calculation of the total SiH_4 dissociation in the discharge, while at the same time the film growth rate was measured using laser reflectance interferometry. The experimental measurements presented here cover a wide range of discharge power (11 mW/cm^2 - 162 mW/cm^2), excitation frequencies (13.56 MHz - 50 MHz), SiH_4 fractions in the SiH_4/H_2 mixtures (2% - 6%) and total gas pressures (0.5 Torr and 1 Torr), in an effort to reveal a possible relation between silane dissociation rate and the film growth rate.

Furthermore, these measurements were combined with a mass transfer model of SiH_4/H_2 discharges that involves gas phase chemistry and plasma surface interactions [14]. The application of the model on the above-mentioned discharge conditions is used to outline the main paths of SiH_4 dissociation in the discharge and to determine the rate of silane dissociation towards neutral radicals and ions. The results presented here indicate a linear relation between the measured film growth rate and the predicted silane dissociation rate, independent of the applied power, frequency or silane partial pressure. The only discharge parameter that seems to affect this relation is the total gas pressure, indicating that at higher pressures the fraction of silicon atoms resulting from silane dissociation that are incorporated to the growing film increases.

2. EXPERIMENTAL

Film deposition studies have been performed in a capacitively coupled Ultra High Vacuum (UHV) parallel plate reactor, with a base vacuum of 10^{-9} mbar. The reactor is equipped with a load lock system for the transportation of the substrates and with four quartz windows suitable for spectroscopic observations. The grounded (deposition) electrode with 90mm diameter is mounted on an ultra high vacuum linear motion feedthrough, allowing the variation of the interelectrode space. In the present study the distance between the two electrodes was fixed at 1.7cm and at 1.5cm for total gas pressures of 0.5Torr and 1Torr respectively. In all cases 2%-6% SiH_4 in H_2 have been delivered in the reactor trough a showerhead-powered electrode. Pressure and flow rate, have been independently adjusted by a downstream throttle valve controller and an upstream mass flow controller, respectively. Glass Corning 7059 has been used as substrate for film deposition, at temperature of 250° C. Laser Reflectance Interferometry (LRI) has been used for the in-situ measurements of the deposition rate.

The real power consumed in the discharge was measured using Fourier transform voltage and current analysis. The excitation voltage and the discharge current waveforms on the powered electrode lead were acquired using a high impedance 1:100 attenuation voltage probe and an 0.1Ω transfer impedance rf current probe. The method for the measurement of the power consumed in 13.56MHz discharges and the extension to include higher frequencies have been presented in detail elsewhere [13,15].

Mass spectrometric measurements have been performed using a Hidden Analytical (type HAL 301) quadrupole mass spectrometer connected at the exhaust port of the reactor. The gas sampled, through a variable leak valve in a small chamber and has been analyzed after 70eV electron impact ionization. The partial pressure of SiH_4 in the reactor has been determined using the ion current $I(m/e)$, at m/e=32 in order to minimize the contribution of higher homologues [16]. The method used for the calibration and the transformation of SiH_4 partial pressures in the sampling chamber to the corresponding partial pressures in the reactor have been described in detail elsewhere [8].

Particle formation has been avoided in all measurements reported here, as monitored by a laser light scattering technique [17].

3. RESULTS AND DISCUSSION

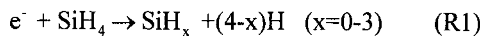
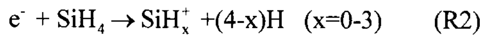
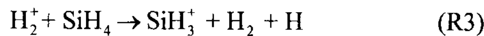
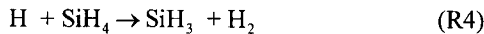
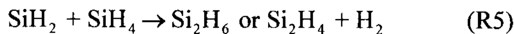
The experimental results presented here deal with the effect of the main SiH_4/H_2 discharge parameters affecting the deposition process of μc-Si:H thin films. More specifically, the effect of excitation frequency, discharge power and silane partial pressure on the total SiH_4 consumption and the deposition rate of μc-Si:H films has been investigated at two total gas pressures of 0.5 and 1Torr. The investigation has been performed by varying one of the above mentioned discharge parameters while maintaining constant all the other discharge conditions. The substrate temperature and the total gas flow rate have not been included in this analysis and in all experiments had constant values of 250 °C and 100 sccm respectively. The electrode gap has been set to the value of 1.7 cm for all measurements carried out at 0.5 Torr and at 1.5 cm for the measurements at 1 Torr, because these settings were found to optimize the deposition rate at the respective gas pressures [18]. The discharge conditions, where power, mass spectrometric and deposition rate measurements were performed, are summarized in Table I.

Table I: Experimental conditions used for the study of the effect of frequency, power and silane partial pressure on the deposition process of microcrystalline silicon at the total gas pressures of 0.5 Torr and 1 Torr.

Discharge parameters	Power	Power	Frequency	SiH_4/H_2 Flow rate	SiH_4/H_2 Flow rate
Pressure (Torr)	0.5	1	0.5	0.5	1
Temperature (°C)	250	250	250	250	250
Electrode gap (cm)	1.7	1.5	1.7	1.7	1.5
Frequency (MHz)	30	30	13.56-30-35-50	30	30
Power (mW/cm2)	67-92-124-162	11-36-50-63	28	48	120
Total flow (sccm)	100	100	100	100	100
SiH_4 flow (sccm)	2	6	2	2-3-4-6	2-3-4-6
H_2 flow (sccm)	98	94	98	98-97-96-94	98-97-96-94

The wide range of discharge conditions that have been applied, leads to a rather extensive range of film deposition rates (0.5 Å/sec - 9.5 Å/sec) and % SiH_4 consumptions (12% - 85%). The mass-spectrometric measurements of the % SiH_4 consumption have been used in the SiH_4/H_2 discharge model, in order to distinguish the different paths of silane dissociation. The discharge simulator includes a gas phase as well as a surface chemistry model as presented in detail in Ref. [14]. The basic output parameters of the model are the rate of production of the effective electron population for SiH_4 dissociation, the radicals flux towards surfaces and the film growth rate.

The application of the model to the present experimental conditions permits the calculation of k_d, while it reveals that the main reactions leading to the dissociation of SiH_4 in SIH_4/H_2 discharges are the following:


$$e^- + SiH_4 \rightarrow SiH_x + (4-x)H \quad (x=0\text{-}3) \qquad (R1)$$

$$e^- + SiH_4 \rightarrow SiH_x^+ + (4-x)H \quad (x=0\text{-}3) \qquad (R2)$$

$$H_2^+ + SiH_4 \rightarrow SiH_3^+ + H_2 + H \qquad (R3)$$

$$H + SiH_4 \rightarrow SiH_3 + H_2 \qquad (R4)$$

$$SiH_2 + SiH_4 \rightarrow Si_2H_6 \text{ or } Si_2H_4 + H_2 \qquad (R5)$$

The relative weight of each of these reactions in SiH_4 dissociation depends of course on the discharge conditions however in all cases, the electron impact dissociation towards neutral radicals (R1) is the main path of SiH_4 consumption. Additionally, R1 and R2 are also the necessary initial steps for any SiH_4 dissociation through any secondary gas phase reactions. The charge transfer reaction (R3) between hydrogen ions and SiH_4 is also important, as it is extremely fast [19], its contribution depending on the degree of H_2 ionization. It is worth noticing that R4 and R5 are related to R1 i.e the enhancement of SiH_4 electron induced dissociation will

result in an analogous enhancement of R4 and R5. This is because atomic hydrogen and SiH_2 that participate in R4 and R5 result from SiH_4 dissociation through R1. Atomic hydrogen is also produced via electron impact dissociation of H_2 which can also be expressed in terms of e^- / SiH_4 dissociation, as both these processes share the same effective electron population. This is true because the two processes have almost the same energy threshold (8.4 and 8.9 eV respectively) and cross sections that have a similar shape in the electron energy range between 8eV and 18eV [20,21]. On the other hand, the relation between R1 and R2, R3 is not so simple, as they depend on electron - molecule collision processes having different electron energy requirements and cross sections. Namely, R1 requires electrons with energy above 8.4eV, R2 electrons with energy above 11.4eV and the production of hydrogen ions that participate in R3 electrons with energy above 15.5eV.

Taking into account the above discussion, the relationship between the total SiH_4 electron-induced dissociation rate and the total SiH_4 dissociation rate will reflect the importance of R1 on SiH_4 consumption. Thus, in fig. 1 and 2 is presented the rate of SiH_4 dissociation through R1 as a function of the total silane consumption rate at the total gas pressures of 0.5 Torr and 1 Torr respectively. The rate of the electron impact silane dissociation ($E.D_{SiH4}$) has been calculated from the product of k_d times the SiH_4 density, while the total silane consumption rate ($T.D_{SiH4}$) has been extracted directly from the mass spectroscopic measurements using the expression [8]:

$$T.D_{SiH_4} = F \times (X_o - X_D) \times N_L, \tag{1}$$

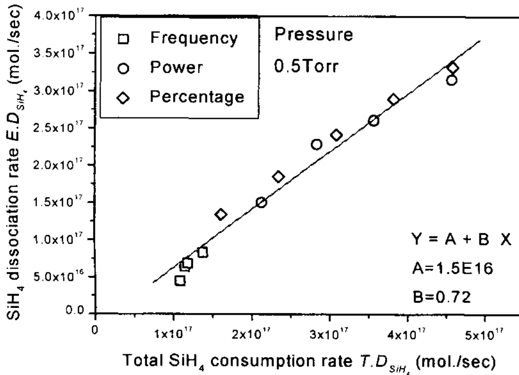


Figure 1: The rate of SiH_4 electron induced dissociation rate as a function of the total silane consumption rate at the pressure of 0.5 Torr.

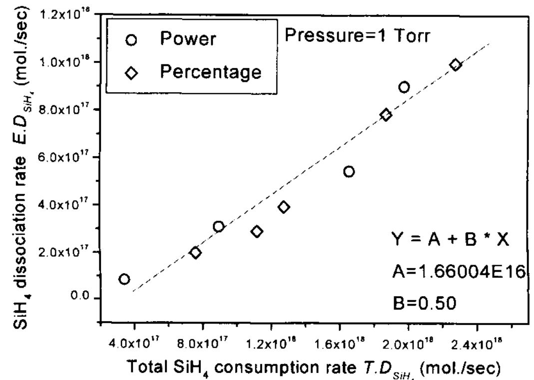


Figure 2: SiH_4 primary dissociation rate as a function of the total silane consumption rate at the total pressure of 1 Torr.

where F is the total gas mixture flow rate, X_o, X_d the fraction of SiH_4 in the discharge off and on conditions and N_L the Loschmidt number.

In both pressures the relation between $E.D_{SiH4}$ and $T.D_{SiH4}$ can be rather well described by a linear expression of the form $E.D_{SiH4} = A + B \times T.D_{SiH4}$, the coefficient B reflecting the relative importance of electron induced dissociation to the total SiH_4 consumption. In the pressure of 0.5 Torr (fig.1), where results of frequency, power and silane partial pressure are included, the linear fitting is fairly good and reveals that R1 is responsible for more than 70% of the total SiH_4 consumption (B=0.72). On the other hand, in the total SiH_4/H_2 pressure of 1 Torr (fig.2), the linear fit is less accurate but within an error limit of about 5%, the relative importance of R1 to the total silane consumption has been found to drop to 50% (B=0.5). This drop indicates that the importance of the secondary gas phase reactions (R4 and R5) is enhanced, as expected, with the increase of pressure.

The change of the relative importance of primary and secondary reactions at the two pressures is also expected to affect the film growth rate. This is because the reactions in the gas phase are responsible for the production of the film precursors and in combination to the mechanism of mass transport of the radicals in the discharge, determine the composition of the radicals flux towards the growing surface. The increase of pressure alters both gas phase chemistry and diffusion of species and this will affect the film growth.

In order to investigate the relationship between the film growth rate and the silane primary dissociation rate ($E.D_{SiH4}$), in fig. 3 and 4 is presented the rate that silicon atoms incorporate to the growing film (S.DR) and the corresponding values of the deposition rate (DR) as a function of $E.D_{SiH4}$ for the total pressures of 0.5 and 1 Torr respectively. The values of S.DR are calculated from the measured values of the deposition rate, using the expression

$$S.DR = DR \times A \times \frac{\rho \times N_A}{m} \tag{2}$$

where A is the grounded electrode area, m the molar mass of silicon atoms and ρ the mass density of μc-Si:H films, while N_A is Avogadro' s number.

In both pressures (fig.3, 4) the relation between the rate that silicon atoms incorporate to the growing film and the SiH_4 primary dissociation rate can be described by a linear expression of the form S.DR= C + D x

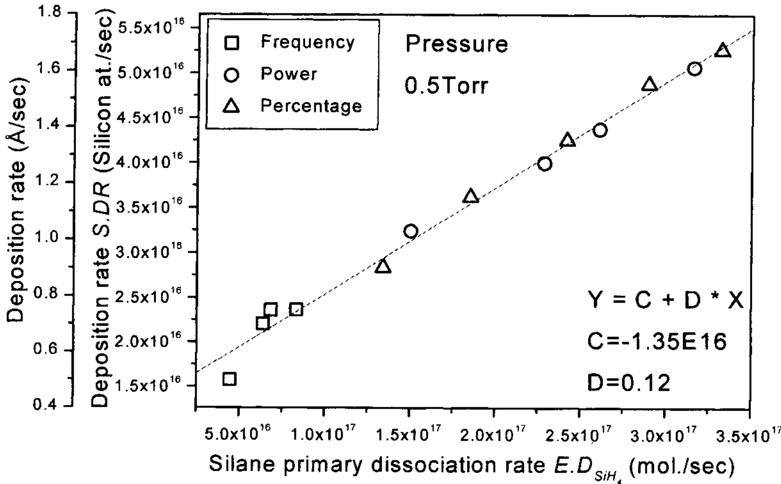


Figure 3: The rate that silicon atoms incorporate to the growing film (S.DR) and the deposition rate as a function of the silane electron impact dissociation rate at 0.5 Torr

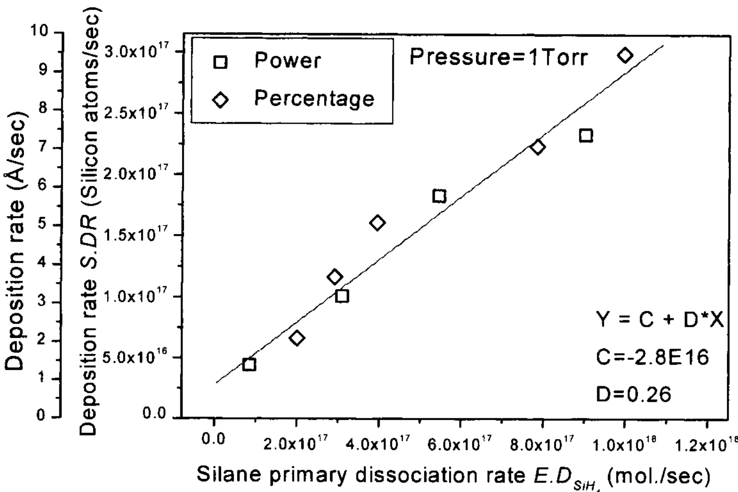


Figure 4: The rate that silicon atoms incorporate to the film growth (S.DR) and the deposition rate as a function of the silane electron impact dissociation rate at the gas pressure of 1 Torr.

$E.D_{SiH4}$. This linear behavior can be understood by taking into account the mechanisms leading to the film growth. More precisely, the measured deposition rate depends on the radicals flux towards the surface and

also on their relative probability to incorporate to the growing film network:

$$DR = 10^8 \frac{\rho}{m}\left(\sum_n a_n s_n D_n \frac{\vartheta N_n}{\vartheta x}\right), \tag{3}$$

where α_n is the number of silicon atoms in radical n and s_n the radical n incorporation probability. The term $D_n(\vartheta N_n/\vartheta x)$ represents the diffusive flux of radicals towards the substrate and according to Gallagher[22] can be expressed as:

$$D_n \frac{\vartheta N_n}{\vartheta x}\bigg|_{x=d} = \frac{u_{th,n}}{2}\frac{\beta_n}{(2-\beta_n)} N_n\Big|_{x=d}, \tag{4}$$

where N_n^d is radical n density at a distance of one mean free path from the substrate, $u_{th,n}$ is the thermal velocity of radicals and β_n the radical loss probability to the surface.

Furthermore, the combination of eqs.(4) and (5) reveals that the film growth rate DR is proportional to the density of radicals near the surface. Thus, with the assumption that the incorporation and loss probabilities s_n and β_n of the radicals do not significantly change under the present experimental conditions, the linear expression between DR and $E.D_{SiH4}$ that has been presented in fig.3 and fig.4, expresses the proportionality between the average SiH_4 dissociation rate and the density of radicals close to the surface. In order to investigate the conditions that lead to such a relation, the density continuity equation of mono-silicon radicals,

$$D_n \frac{\vartheta^2 N_n}{\vartheta x^2} + k_{\sec} N_n N_{SiH4} + k_{\sec} N_n^2 = E.D_{SiH4} \frac{d \times I(x)}{I_{TOT}} \tag{5}$$

and of silicon dimmers

$$D_{2n} \frac{\vartheta^2 N_n}{\vartheta x^2} + k_{2\sec} N_{2n} N_{SiH4} + k_{\sec} N_{2n}^2 = k_{\sec} N_n N_{SiH4} + k_{\sec} N_n^2, \tag{6}$$

at every point between the two electrodes can be used. In eqs. (5) and (6) the left hand terms represent the depletion of these radicals through diffusive losses to the surfaces and through secondary gas phase reactions while the right hand terms stand for the production of these radicals through silane electron impact dissociation and/or secondary reactions. The average SiH_4 dissociation rate $E.D_{SiH4}$ is inserted in the density continuity equations by the term $d \times I(x)/I_{TOT}$ that represents the normalized SiH_4 dissociation rate profile in the space between the two electrodes.

Furthermore, integration of eqs.(5) and (6) from the point of maximum density of radicals $x_n^{\max}$ to the deposition electrode d, rearrangement and elimination of the terms involving the less important radical-radical reactions, leads to the following expressions for the relation between the $E.D_{SiH4}$ and the density close to the surface, for mono silicon radicals:

$$N_n\Big|_{x=d} = \frac{E.D_{SiH4} d \times \int_{x_n^{\max}}^{d} I(x)/I_{TOT} dx}{u_{th,n}\beta_n/[2(2-\beta_n)]} - \frac{\left[k_{\sec} N_{SiH4} \overline{N}_n (d - x_o^{\max}) \int_{x_o^{\max}}^{d} (I_n(x)/I_{n,TOT}) dx\right]}{u_{th,n}\beta_n/[2(2-\beta_n)]} \tag{7}$$

and for silicon dimmers:

$$N_{2n}\Big|_{x=d} = \frac{E.D_{SiH4} d \times \int_{x_n^{\max}}^{d} I(x)/I_{TOT}}{u_{th,2n}\beta_{2n}/[2(2-\beta_{2n})]} - \frac{\overline{N}_n u_{th,n}\beta_n/[2(2-\beta_n)]}{u_{th,2n}\beta_{2n}/[2(2-\beta_{2n})]} - \frac{k_{\sec} N_{SiH4} \overline{N}_{2n} (d - x_o^{\max}) \int_{x_n^{\max}}^{d} I_n(x)/I_{TOT}}{u_{th,2n}\beta_{2n}/[2(2-\beta_{2n})]}. \tag{8}$$

Equations (7) and (8), reveal that the density of radicals near the growing surface is not only a function of $E.D_{SiH4}$ but also depends on the SiH_4 density N_{SiH4}, the average density of radicals between $x_o^{\max}$ to d $\overline{N}_n$, $\overline{N}_{2n}$ and the spatial distribution of both silane dissociation rate $\int_{x_n^{\max}}^{d} I(x)/I_{TOT}$ and radicals density

$\int_{x_n^{max}}^{d} I_n(x)/I_{TOT}$. All these terms, except the spatial distribution of the SiH_4 dissociation rate, are involved in the second term of the right hand part of eq. (7) and (8), while the fact that they do not disturb the linear relation between DR and $E.D_{SiH4}$ indicates that these terms are of minor importance compared to the

$$\frac{E.D_{SiH4} d \times \int_{x_n^{max}}^{d} I(x)/I_{TOT} dx}{u_{th,2n}\beta_{2n}/[2(2-\beta_{2n})]}$$

term. In particular, the linear fit that is shown in figs. 3 and 4., includes all these variables in the constant term C which is in both pressures negative and quite lower than the S.DR.

The most important feature of eqs. (7) and (8) is that the linear relationship of *DR* and $E.D_{SiH4}$, strongly depends on the shape of the profile of silane dissociation rate $\left(\int_{x_n^{max}}^{d} I(x)/I_{TOT} dx\right)$. Thus, a discharge parameter that will affect the spatial distribution of silane dissociation rate will consequently also disturb the linear relation. From the discharge parameters presented here, the increase of silane partial pressure and the increase of power is not expected to affect the discharge structure and consequently the spatial production of radicals, leading to the observed linear relation. In addition, excitation frequency (fig.3), which is well known to affect the spatial production of species[15], does not significantly affect the linear relation, however this is mainly due to the quite low values of either *DR* or $E.D_{SiH4}$.

Total gas pressure is also a discharge parameter that alters the spatial distribution of silane dissociation rate and this is reflected in fig.3 and fig.4. Namely, although in both pressures the dependence of DR on $E.D_{SiH4}$ is linear, the slope changes from 0.12 to 0.26 as pressure increases from 0.5 Torr to 1 Torr. This change indicates that in the low-pressure case, from the silicon hydrides produced via electron impact SiH_4 dissociation, a fraction of about 12% is finally incorporated to the film in the deposition electrode. On the other hand, in the high-pressure case (fig. 4) the deposition efficiency increases and this fraction takes the value of 26%.

The increase of the deposition efficiency with pressure is the result of the enhancement of secondary gas phase reactions that can in some cases, produce radicals with two or more silicon atoms (R4, right term of eq.(6)). The incorporation of these radicals to the film growth will give a rise to the deposition efficiency and the deposition rate as they offer two or more silicon atoms to the silicon network (eq.(4), α_n). On the other hand, in the low-pressure case secondary gas phase reactions are not favored and the radicals that mainly contribute to the film growth are mainly mono-silicon radicals. This observation indicates that the increase of pressure in combination to small electrode gaps is necessary when a rapid growth process is required.

4. CONCLUSIONS

The relation between electron impact and total dissociation rate of silane with the film growth rate, has been investigated by applying a combination of experimental measurements and modeling of gas phase/surface chemistry of highly diluted SiH_4 in H_2 discharges. A wide range of frequencies (13.56 MHz – 50 MHz), power densities (11 mW/cm^2 – 162 mW/cm^2) and silane partial pressures (2% - 6%) at two total gas pressures of 0.5 Torr and 1 Torr has been studied, in an effort to generalize the conclusions as much as possible.

A linear dependence of the rate of SiH_4 primary dissociation on the total silane dissociation rate has been revealed that is independent of power, silane partial pressure and frequency and is only affected by the increase of the total pressure. This linear relation indicates that in the case of 0.5 Torr, SiH_4 electron impact dissociation is responsible for 70% of the total silane consumption, while the increase of the total pressure induces a drop of its relative importance to about 50%.

The relation between the measured film growth rate and the silane primary dissociation rate has also been

found to be linear, independent of the discharge conditions. The only discharge parameter that affects this relation is again the total gas pressure, indicating that at higher pressures the fraction of silicon atoms that are incorporated to the film growth relative to the silicon atoms that result from primary silane dissociation increases. This increase has been attributed to the production of additional two-silicon atoms precursors through secondary gas phase reactions.

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