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THE EFFECT OF IONS ON THE GROWTH OF AMORPHOUS AND MICROCRYSTALLINE SILICON*

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We present an overview of the quantification of the ions in the growth process of both amorphous (a-Si:H) and microcrystalline silicon (μc-Si:H). In the deposition of a-Si:H the ion energies are typically in the 20-30 eV range. The fluxes relative to the growth rate are typically 25%. However, the actual contribution to growth is 5-10%. The ion bombardment expressed in ion kinetic energy per deposited atom is an important parameter in the resulting material structure. The amount of energy that is needed to create a dense network decreases with increasing substrate temperature, thereby indicating the interexchangebility of these two parameters. The deposition of microcrystalline silicon by RF-PECVD is, due to the high ionization potential of hydrogen with respect to that of silane, dominated by ions instead of by radicals as in the deposition of a-Si:H. This implies the need of considering ions in the growth models of microcrystalline silicon. Ion mass spectra show the presence of polymerisation reactions of ions in the gas phase. It appears that ions with 3 or 4 silicon atoms are the most dominant species present. Detailed investigation of the amount of hydrogen on ions with 6 or more Si atoms indicate the possible presence of ring structured silicon ions.

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1 Introduction

Hydrogenated amorphous silicon (a-Si:H) and microcrystalline silicon (μc-Si:H) are used in thin film transistors (TFT) to drive pixels in LCD screens as well as in solar cells. Both these materials are deposited commonly from silane (SiH_4) hydrogen (H_2) mixtures by means of radio frequent plasma enhanced chemical vapour deposition (RF-PECVD). In this process the gas is dissociated and ionized by means of electrons that gain their energy in the oscillating electric field applied over two parallel electrodes. The produced radicals diffuse towards the electrodes, whereas the created ions are accelerated in the sheath regions and drift towards the electrodes. Both the radicals as well as the ions have a possibility to contribute to the film growth.

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The deposition of good quality hydrogenated amorphous silicon is believed to proceed by a dominant contribution of the SiH_3 radical [1]. The ion contribution to the growth is in general low, however the importance of the high kinetic energies that these species may have, makes them nevertheless important for the growth process.

In this paper we will address the contribution of ions to the growth process of both amorphous and microcrystalline silicon, and place this in relation with the structural material properties. Hereto we will discuss the kinetic energies of the ions at the substrate, the kind of ions reaching the substrate, the ion flux and quantify the contribution of the ions to the growth rate. These quantities allow us to study the relation between ion 'bombardment' and structural material properties as density, hydrogen bonding configuration and stress.

2 Experimental method

The ions are studied by means of an ion energy and mass spectrometer located behind the grounded substrate. The ions are sampled through a 30 μm diameter orifice. Mass resolved ion energy distribution functions (MRIEDF) are used to determine the plasma potential and the charge carrier density in the sheath. This latter is possible due to charge exchange processes in the sheath of the RF plasma, resulting in characteristic peaks in the MRIEDF. Combination of these two quantities allows estimation for the ion flux. The method is described extensively elsewhere [2].

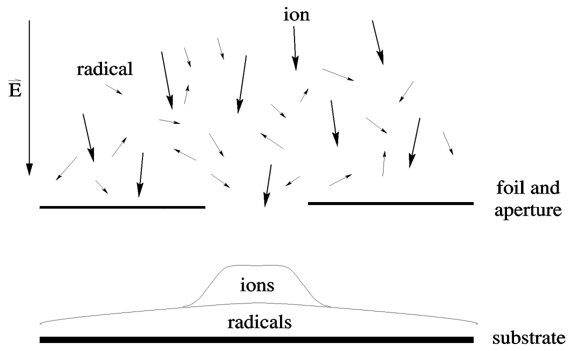

Fig. 1. Schematic diagram of the deposition profile of both radicals and ions behind an aperture.

The ion contribution to the growth is determined from the partial spatial separation of ions and radicals (PSSIR) behind an aperture. This is due to the different angular distribution of the velocity of radicals, which diffuse through the sheath, and the ions, which are accelerated towards the substrate. The principle is indicated schematically in Fig. 1. The radicals deposit on the substrate far away from the aperture, whereas the ions deposit mostly directly behind the aperture. The resulting thickness profile is measured with a surface profiler. A model [3, 4] is used to determine the surface reaction probability of the radicals, the relative ion contribution and the width of the angular distributions of the ion velocities.

The samples are deposited on Corning 7059 glass and c-Si. Reflection-transmission mea-

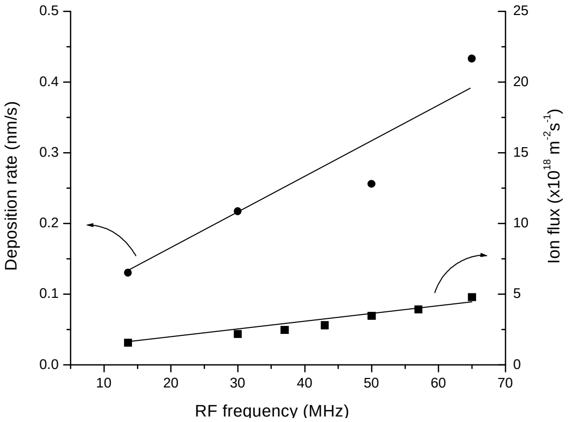

Fig. 2. The growth rate r_d and ion flux as function of the RF frequency. The right hand axis is scaled to the left hand side axis as is explained in the text. Lines are guides to the eye.

surements give the thickness of the film as well as the optical constants. The hydrogen- bonding configuration is determined from infrared transmission measurements. The microstructure parameter R^* is defined as

$$R^* = I_{2100}/(I_{2000} + I_{2100}),$$

where I_{2000} is the integrated absorbance of the Si-H band at 2000 cm^{-1} and I_{2100} the integrated absorbance of the Si-H_2 band at 2100 cm^{-1}. Material with good opto-electronic properties is generally characterized by an R^* smaller than 0.1. The stress in the films follows from the difference in curvature of the c-Si substrate before and after deposition of the film [5].

3 Results

3.1 Ion energies and fluxes

In order to study the ion bombardment the energy of the ions and their flux has to be quantified. A measurement series in which the RF frequency is changed is very illustrative and will be discussed in the following.

By many groups higher RF frequencies than the conventional 13.56 MHz is used to increase the deposition rate [6, 7]. In Fig. 2 we plotted the growth rate as a function of the frequency. In going from 13.56 MHz to 65 MHz the growth rate is seen to increase by a factor of 4. The growth rate is measured in the middle of the grounded electrode.

The typical plasma potential is 20 to 25 V. This voltage is independent of frequency and its low value is due to the asymmetric configuration of the reactor: the grounded electrode is about a factor of 2 larger than the powered electrode. MRIEDF measurements show however that the charge carrier density increases with applied RF frequency. As a consequence the sheath thickness decreases, as is also observed by others [8], and the ion flux increases. The ion flux is also plotted in Fig. 2. The left hand scale, the deposition rate, and the right hand scale, the flux, are scaled to each other by using the atomic density of c-Si, 5×10^{28} m^{-3}, and assuming a unity sticking probability of the ions.

It is seen that *if* all the ions stick, they contribute about 25% of the silicon atoms to the growth. The contribution of Si by the ions to the growth is addressed in more detail below.

One remarkable observation is the fact that the amount of silane that is being used in the plasma, as determined from mass spectrometry measurements, is independent of frequency, and amounts to 4.0±0.4 sccm (standard cubic cm per minute), while the growth rate increases. This may be explained as follows. The DC-bias voltage V_{dc} that forms on the powered electrode changes from -124 V at 13.56 MHz, to only -8.8 V at 65 MHz. At the same time the plasma potential remains constant. This has to imply that the RF voltage decreases with frequency, as is indeed reported in literature [9]. As a consequence the voltage drop over the sheath in front of the grounded electrode, V_{pl}, and the voltage drop over the sheath in front of the powered electrode, namely $V_{\mathrm{pl}} + V_{\mathrm{dc}}$, become comparable in magnitude. Thus discharge becomes more symmetrical at higher frequencies. This suggests that also the deposition will become more evenly distributed over the reactor. This suggestion is supported by the fact that based on a homogeneous deposition on the walls of the reactor and the measured depletion we would expect a growth rate of 0.44 nm/s. This growth rate is measured at the higher frequencies, but lacks at the lower frequencies.

In other measurements we also find that the plasma potential is in the 20 to 30 V range. These values are also found in plasmas with other gases as e. g. Ar or H_2 [7, 10, 11]. The ion fluxes are typically around 1×10^{18} $m^{-2}s^{-1}$ and may explain 20% of the growth rate if all ions stick and contribute 1 silicon atom to the growth.

3.2 Ion bombardment and material structure

The effect of the ions on the material structure is best seen in the following. The series that we present here is generated in silane-argon mixtures. The conditions are a RF frequency of 50 MHz, a pressure of 40 Pa, and flows of 30 sccm SiH_4, and 60 sccm Ar. The power is varied from 3 to 50 W. Between 6 and 12 W, the plasma changes from the dust free α regime to the dust producing γ' regime. In the experiments, this transition is observed by the change in impedance of the plasma and also by the visual impression of the plasma. In the transition from the α to the γ' regime, the optical emission from the plasma becomes brighter and more homogeneously distributed. In the α regime the brightest parts of the plasma are at the boundary between plasma bulk and sheaths. The transition is also observed in the *decrease* of the absolute magnitude of the DC self-bias. While normally the DC self bias voltage is seen to increase with increasing power, the observed decrease is due to the transition to the γ' regime. In the transition the enhancement of the growth rate is larger than the change in ion flux. As a result the relative contribution of ions to the growth diminishes. Whereas the ratio between ion flux and deposited Si flux is typically 25% in the α regime, this ratio is only 10% in the γ' regime.

The DC self bias is very low in the γ' regime, typically between -2 and -1 V. The plasma potential increases with increasing power and tends to saturate at a value of 30 V at the highest powers. The growth rate, plotted in Fig. 3a, reaches its maximum around 30 W and decreases somewhat at higher powers. The microstructure parameter, see Fig. 3b, is low in the material deposited at 3 W. It increases rapidly with increasing power, reaches a maximum at a power of 20 W, and then decreases. A high value of the microstructure indicates a relatively large fraction

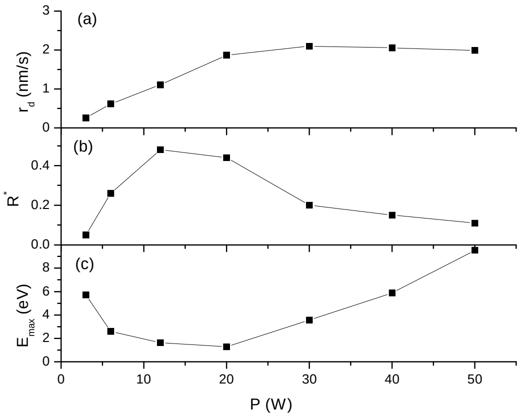

Fig. 3. The growth rate r_d (a), the microstructure parameter R^* (b), and the kinetic ion energy per deposited atom E_{max} (c) as function of power in a silane-argon plasma.

of Si-H_2 bonds in the material, corresponding to an open material structure.

Andújar *et al.* [12] studied the influence of power and pressure on the material quality in both the α and γ' regime in pure silane plasmas at 13.56 MHz. The series we presented here exhibit the same trends in material properties as the materials they studied. The microstructure increases with power when entering the γ' regime, but decreases again towards the higher powers. Such a behaviour is in fact also observed in pure silane plasmas at 50 MHz and was reported by Meiling *et al.* [13]. Nishikawa *et al.* [14] observed an amelioration of the electrical properties of the deposited films at high RF power densities.[1] In the literature it has been suggested that the decrease of the microstructure parameter at the highest powers is related to an increased ion bombardment [12, 15].

In ion beam assisted deposition the ion bombardment is quantified by the amount of kinetic energy that is transferred by the *ions* per deposited *atom* [16]. We denote this quantity by E_{max} and deduce it from the plasma potential V_{pl}, the ion flux Γ, the atomic density of the deposited network ρ, and the growth rate r_{d}: $E_{\mathrm{max}} = V_{\mathrm{pl}}\Gamma/(\rho r_{\mathrm{d}})$. This quantity thus has the dimensions of eV per deposited atom.

In order to demonstrate that the suggestion of ion bombardment (=E_{max}) is appropriate to explain the behaviour of the structural properties of the a-Si:H, we have plotted the ion kinetic energy per deposited atom E_{max} as function of the power in Fig. 3c. At 3 W the value of E_{max} is 5.7 eV, it reaches a minimum of about 1.5 eV around 20 W, and subsequently increases again. The microstructure parameter is highest where E_{max} is lowest. When E_{max} increases, R^* decreases while the growth rate remains constant. This observation is a strong indication that E_{max} is an important parameter in describing the microstructure parameter. We supported

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[1] Both the dark- and photo-conductivity of our sample deposited at 40 W have been measured. The photo-conductivity has been measured under AM1.5 conditions. The dark- and photo- conductivity are $1 \times 10^{-10}\Omega^{-1}\mathrm{cm}^{-1}$ and $1.2 \times 10^{-4}\Omega^{-1}\mathrm{cm}^{-1}$, respectively. These values are indicating good electronic properties, e. g. a low defect density and a high carrier mobility.

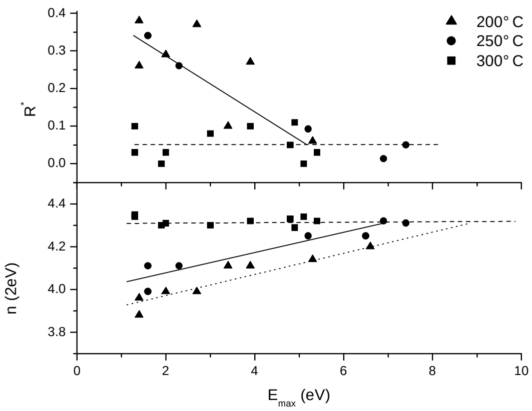

Fig. 4. Material properties as function of $E_{\max}$ at three substrate temperatures: (a) the microstructure parameter R^* and (b) the refractive index n at a photon energy of 2.0 eV.

this vision more extensively elsewhere [2]. There is a one-to-one relation between the relative strength of the ion bombardment, expressed in terms of $E_{\max}$, and the microstructure parameter. At a substrate temperature of 250 °C, at least 5 eV ion kinetic energy is needed to form a dense material structure.

Now that we have seen the influence of ion bombardment ($= E_{\max}$) on the material, we will investigate whether ion bombardment and substrate temperature may be interchanged. Hereto we studied the influence of ions at three different substrate temperatures. We compare three series in which the process pressure was changed. The series at 250 °C has been discussed extensively elsewhere [2]. In Fig. 4 we plot the microstructure parameter and the refractive index as function of $E_{\max}$. The microstructure parameter R^* shows the same behaviour versus the ion energy $E_{\max}$ at substrate temperatures of 200 and 250 °C. However, at 300 °C the value of R^* is less than 0.1 in the measured range, so at least down to $E_{\max} = 1.1$ eV. In the samples deposited at a substrate temperature of 200 °C the refractive index appears to be lower at every $E_{\max}$ than at a substrate temperature of 250 °C. The threshold to make a dense material is observed to be a few eV higher than in the 250 °C series. The refractive index in the 300 °C series is independent of $E_{\max}$ and is high.

Another structural parameter that is studied is the internal stress in the a-Si:H film. The relation between stress and $E_{\max}$ is plotted in Fig. 5. The points of the 200 and 250 °C substrate temperature series show the same relation, the stress increases linearly with $E_{\max}$. The data points of the 300 °C substrate temperature series follow the same slope, but exhibit a 600 MPa higher compressive stress at given $E_{\max}$. A compressive stress of 600 MPa corresponds to a compression of about 1%, due to an excess of about 1% of Si-atoms in the network. The films that were deposited at 300 °C under conditions where the value of $E_{\max}$ was larger than 4.5 eV started to peel off from the silicon substrate directly after exposure to air. The circle in the figure surrounds the corresponding data points. As a result of the peeling, stress relief occurred and the stress is lower than in the as-deposited state. Peeling off of a-Si:H layers with a stress of about

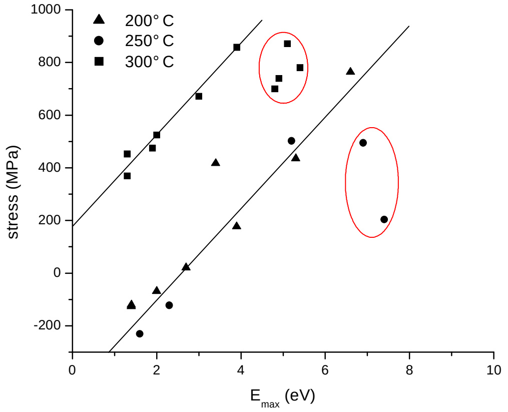

Fig. 5. Internal stress as function of $E_{\rm max}$ in the pressure series at 50 MHz. Plotted are the results for three different temperatures. Lines are guides to the eye.

1 GPa is quite common. We suggest that the samples that are deposited at 250 °C with $E_{\rm max}$ higher than 6.5 eV also already peeled on a microscopic scale (so not visible with the naked eye), and that therefore their stress is lower than the extrapolated behaviour from lower values of $E_{\rm max}$. The stress of these films would be comparable to the stress in the films at 300 °C that peeled off.

The effects of ion bombardment on the refractive index, the microstructure parameter, and the stress all indicate that ions are needed to create a dense amorphous silicon network at 200 and 250 °C. At a substrate temperature of 300 °C much lower ion bombardment is needed. Based on the behaviour of the stress a 4 eV/deposited atom lower ion bombardment is needed at 300 °C than at 250 °C to get a similar stress. If we combine this result with the observed behaviour of the microstructure parameter and the refractive index we might conclude that at 300 °C at least 1 eV/deposited atom is needed. So the sample that was deposited with the lowest ion bombardment in that series of 1.1 eV should be close to that limit.

3.3 Ion species bombarding the surface

Ions arriving at the electrode clearly demonstrate the importance of gas phase polymerisation as is also observed by others [17, 18]. In Fig. 6 a typical mass spectrum, taken under typical deposition conditions, is shown. This mass spectrum is corrected for the mass transmission function of the instrument but not for the different isotopes of Si. Ions with x Si atoms have a mass of $28x$ amu. The ions with mass around 44, and multiples of 28 amu higher are $\mathrm{Si}_n\mathrm{OH}_y^+$ ions. Ions with 3 or 4 Si atoms are the most abundant ions present. The energy of the ions corresponds with $eV_{\rm pl}$, so all these ions are originating from the plasma bulk and are therefore formed in exothermic reactions.

In Fig. 7 the relative abundance of various ions with the same amount of Si atoms is depicted. The spectrum is shown as if only ^{28}Si were present. A few general features are evi-

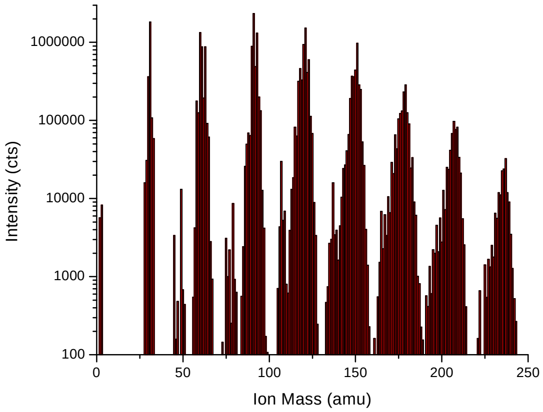

Fig. 6. Ion mass spectrum, corrected for the mass transmission function of the mass spectrometer. The plasma conditions are 10 W RF power, RF frequency of 050 MHz, process pressure of 12 Pa, and flows of 30 sccm SiH_4 and 30 sccm H_2.

dent. $Si_nH_{2n+2}^+$ ions, corresponding to the ionic state of a stable chain neutral (Si_nH_{2n+2}), are never measured. Measurements on ions with 2 to 5 silicon atoms clearly show the existence of $Si_nH_{2n+3}^+$ ions. Such ions are formed as a result of proton attachment on a neutral molecule and are a common feature in plasmas. Well known is e.g. the ions H_3^+ or ArH^+. For the ions with 1 and 3 to 5 silicon atoms the most abundant ion present is $Si_nH_{2n+1}^+$, whereas in the case of two silicon atoms this is $Si_2H_4^+$. All this indicates a polymerisation reaction in which in general a SiH_2 is inserted:

$$Si_nH_{2n+1} + SiH_4 \rightarrow Si_{n+1}H_{2(n+1)+1} + H_2.$$

However in going from 5 to 6 silicon atoms the net result is only the insertion of a Si atom resulting in $Si_6H_{11}^+$ as the most abundant ion with 6 Si atoms. $Si_6H_{13}^+$ and $Si_6H_{15}^+$, corresponding to $Si_nH_{2n+1}^+$ and $Si_nH_{2n+3}^+$, are however at this pressure still present. This H distribution is not incompatible with the existence of ring structured Si ions, since passivated molecules with 6 silicon atoms would contain 12 H. The lack of H on the ions with 7 and 8 silicon atoms compared to the chain like ions might form a support for this statement. Kinetic simulations on clustering of silane in pyrolysis show the formation of ring-like structures [19].

An additional indication for the existence of ring structured ions is by investigating the abundance of the ions with different number of H atoms as a function of the pressure. Fig. 7a shows the ions at 12 Pa, whereas Fig. 7b shows the ions at 50 Pa. When the gas pressure is increased the sheath thickness decreases [2] however the product of sheath thickness and pressure pd_s increases from about 20 Pa mm at 8 Pa to about 90 Pa mm at 50 Pa, thus reflecting more collisions in the sheath. This larger amount of collisions is reflected in the decrease of the abundance of the less stable ions, as for example $Si_nH_{2n+3}^+$ ions, which will be lost in a collision as is known to happen for H_3^+ or ArH^+. The same behaviour is seen for the ions that are certainly related to chain-like structures. The ring-structured ions will be both more stable both in the plasma bulk

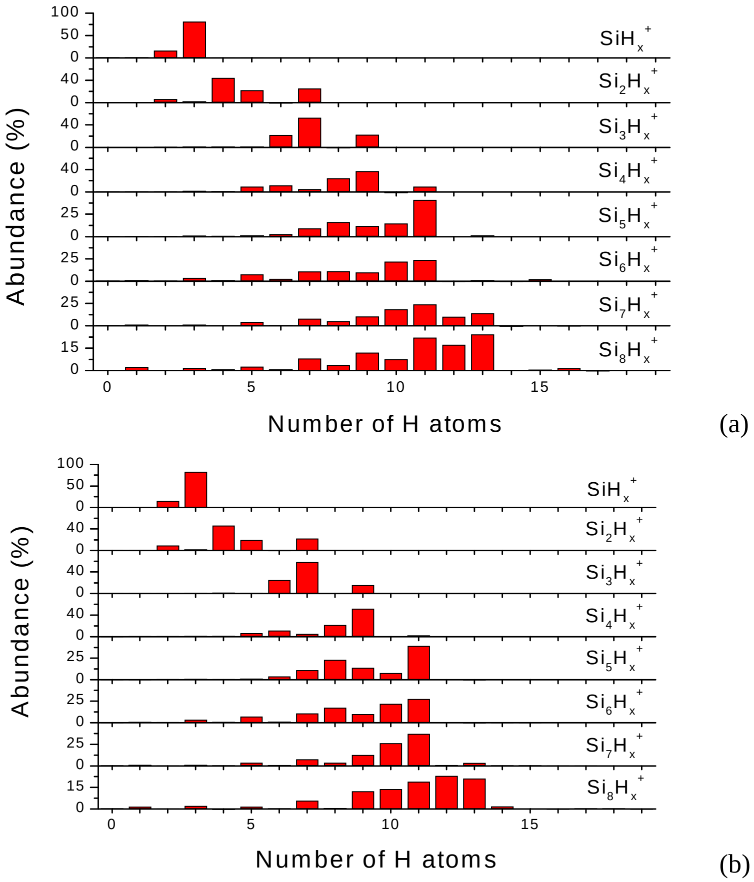

Fig. 7. (a) The relative abundance of ions with different amount of H atoms for the different Si numbers at (a) 12 Pa, and (b) 50 Pa.

and the sheath, since it is harder to break into smaller pieces. Further study should be performed to demonstrate this hypothesis of ring structured ions in silane plasmas.

The relative abundance of the different ion species with the same number of silicon atoms is remarkably constant upon change of the process pressure by a factor 4. Especially if one knows that in going from the α to the γ' regime (dust free-dust producing) many plasma parameters change. The electron temperature increases, the partial pressure of hydrogen increases due to a more efficient power coupling to the plasma, the disilane partial pressure increase, and the plasma potential decreases. The disilane partial pressure is typically 1% of the total pressure. The fact that the relative abundance of the ion species within one order remains the same, demonstrates that the gas phase chemistry does not change. The same silane ions are created and react with the background gases to polymerise. The only difference is that ions tend to become heavier at higher gas pressures.

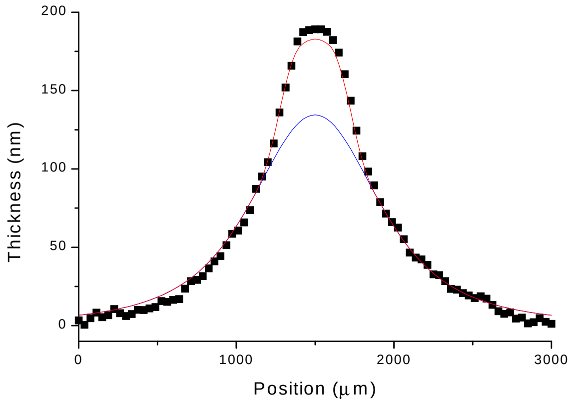

Fig. 8. The deposition profile behind an circular orifice. The wings are completely due to deposition of radicals, while the central part has also a contribution of ions. The surface reaction probability of the radicals is 0.42 and the ion contribution is 4%. The solid lines are the fitted radical (lower curve) and radical plus ion profile (upper curve in central part). For a detailed description of the method see Ref. [3,4].

3.4 Contribution of ions to the growth rate

In section 3.1 we showed that under typical deposition conditions the ion flux is as large as 25% of the total silicon flux needed to achieve the measured growth rates. We used the PSSIR method to quantify the actual contribution of ions to the growth. A typical measurement is shown in Fig. 8. The deposition profile is obtained by depositing through a 460 μm diameter orifice. The lower solid line is the fitted deposition profile due to the radicals. In the central part also ions contribute. The solid line through the measurement points is the fit with both radicals and ions contributing. Under these conditions the ion contribution to the growth is 4.0%. The measurements have been discussed in more detail elsewhere [4]. Typically the contribution of ions to the growth of a-Si:H is in the 5-10% range, which is reasonable when compared with literature [20, 21]. The contribution to the growth is lower than expected based on the ion flux, which might be attributed to a sticking coefficient lower than one for the ions.

The ions are the dominant growth precursors under conditions of microcrystalline growth. This is seen in Fig. 9 where we plotted the total growth rate and the contribution of the ions as function of process pressure. The deposition of μc-Si:H is normally achieved by high hydrogen dilution, typically $>$95% H_2 in order to obtain a high atomic hydrogen flux. A high atomic H flux seems to be needed to form microcrystalline silicon. Although the ions are the dominant species they are not a prerequisite to form microcrystalline silicon as we demonstrated by also studying hot wire CVD [4]. The high ion contribution under high dilution is due to an increasing electron temperature when admixing more hydrogen and the efficient charge exchange process between hydrogen ions and silane molecules. The electron temperature increases since the ionization potential of H_2 is higher than of SiH_4. As a result the plasma will ionize the silane relatively more than hydrogen even in a large surplus of hydrogen present in the source gases. The hydrogen ions that are created and interact with silane are lost in the following charge exchange reaction:

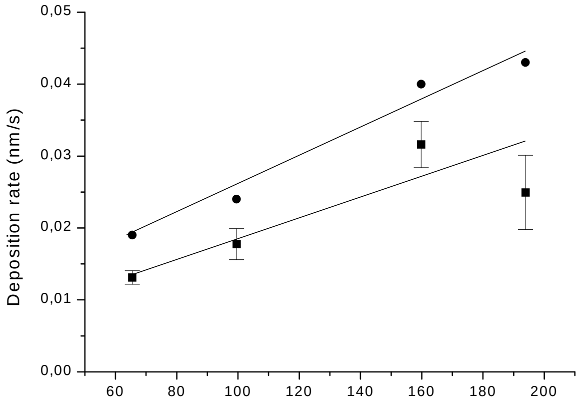

Fig. 9. The growth rate and the growth rate due to the ions as function of process pressure. The circles represent the total growth rate, the squares the growth rate due to the ions. Under these conditions microcrystalline silicon is formed.

H_2^++SiH_4 $\rightarrow$ H_2+SiH_x^+ $+ (4-x)$H. This process is known to have a large cross-section [22]. In our model to determine the ion contribution to the growth, we did not take into account the etching by atomic hydrogen during growth. If this process is important we most probably will overestimate the ion contribution.

The influence of the ions on the resulting material properties in microcrystalline silicon is addressed elsewhere [4].

4 Conclusions

We have presented an overview of the quantification of the ions in the growth process of both amorphous and microcrystalline silicon.

In the deposition of a-Si:H the ion energies are typically in the 20-30 eV range in the system we studied. The fluxes relative to the growth rate are typically 25%. However the actual contribution to growth is 5-10%. The ion kinetic energy per deposited atom is seen to be an important parameter in the resulting material structure. The amount of energy that is needed to create a dense network decreases with increasing substrate temperature, thereby indicating the interexchangebility of these two parameters.

The deposition of microcrystalline silicon by RF-PECVD is, due to the high ionization potential of hydrogen with respect to that of silane, dominated by ions instead of by radicals. This implies the need of considering ions in the growth models of microcrystalline silicon. So far these species have been neglected.

Ion mass spectra show the presence of polymerisation reactions of ions in the gas phase. It appears that ions with 3 or 4 silicon atoms are the most dominant species present. Detailed investigation of the amount of hydrogen on ions with 6 or more Si atoms might indicate the presence of ring structured silicon ions. However more study is needed to confirm this hypothesis.

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