Powder formation in germane–silane plasmas

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The powder formation in a germane (GeH₄)–silane (SiH₄) plasma was investigated for a range of deposition parameters used in the processing of amorphous silicon devices, in particular the pressure, p, and the total gas flow, φ. For that purpose the plasma was monitored continuously by measuring the dc self-bias, Vₛᵇ, as a function of time. At certain plasma conditions oscillations in the self-bias are observed. In literature these oscillations have been related to the spatial oscillation of the powder-forming region in the plasma and they can be used as an indication of the so-called α–γ’ transition of the plasma, i.e., the moment at which powder formation starts to occur in the plasma. Changes in the plasma are monitored by measuring the amplitude of the oscillations of the self-bias, ΔVₛᵇ. It was found that the dependence of ΔVₛᵇ on p can be divided into three ranges: (i) low-pressure range in which ΔVₛᵇ is small; (ii) medium-pressure range in which ΔVₛᵇ varies significantly and powder is formed in the plasma; and (iii) high-pressure range in which the plasma is visually unstable. The pressure at which the transition from range (i) to (ii) occurs is indicative of the α–γ’ transition and appears to depend on the gas composition, in particular the [GeH₄]∕[SiH₄] flow ratio. This ratio also determines the rate of powder formation. We argue that the production of GeH₂ radicals is responsible for this formation. In addition we conclude that the α–γ’ transition is determined by the partial pressure of SiH₄ plus GeH₄ and is not dependent on the residence time of the gas in the reaction chamber. © 2000 American Vacuum Society.

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I. INTRODUCTION

For the deposition of hydrogenated amorphous silicon (a-Si:H) and its alloys, like hydrogenated amorphous silicon germanium (a-Si₁₋ₓGeₓ:H) and carbon (a-Si₁₋ₓCₓ:H), the most widely used and established technique is plasma enhanced chemical vapor deposition (PECVD). Currently, other promising techniques have become available, like hot-wire chemical vapor deposition,¹ with which good and, more importantly, stable material can be made. However, these methods are still under investigation and are not yet applied in industrial processing. The use of the PECVD process has prompted research on this subject, especially in relation to the deposited material.² ³ The PECVD process is very complex and a variation of the process parameters immediately affects the properties of the deposited material. The process becomes even more complicated when other gases are used in addition to the main precursor silane (SiH₄). For the production of solar cell devices a high quality of the individual layers is essential, implying that a good control of the deposition process is necessary. In particular powder formation can be detrimental for the device performance. In this article we aim to find a relation between the occurrence of powder formation in silane–germane plasmas and the process parameters.

Since Carlson et al.⁴ made the first solar cell, developments in cell design and material research have resulted in the use of amorphous silicon alloys. The performance of multijunction cells was improved⁵ by the use of a-Si₁₋ₓGeₓ:H as material for the intrinsic layer of the bottom cells, obtained by adding germane (GeH₄) to the gas mixture. This material has a higher absorption, especially for lower photon energies. Indeed, the incorporation of a-Si₁₋ₓGeₓ:H layers has lead to a triple solar cell with a stabilized conversion efficiency of 13.0%.⁶ At this moment high quality a-Si₁₋ₓGeₓ:H can be produced over a broad deposition temperature range by applying high hydrogen dilution.⁷ ⁸ Thereby problems of so-called thermal damage to other layers can be avoided.⁷

For the production of layers of high-quality material, stable plasmas are important and this requires knowledge about the relation between plasma instabilities and deposition parameters, in particular powder formation in the plasma. In this article we report on the powder formation in SiH₄–GeH₄ plasmas using a range of deposition conditions and applying several hydrogen (H₂) dilutions. Our aim is to find a relationship between the onset and the rate of powder formation on one hand, commonly known as the α–γ’ transition,⁵ and on the other hand the gas composition of SiH₄–GeH₄ plasmas. In the α-regime electrons at the plasma sheath edge near the plasma bulk gain energy from the sheath electric field and are “surfing” on an electric field wave. This has been referred to as collisional “wave-riding.”¹² In the γ’ regime electrons are attached to nanometer size clusters, trapped within the plasma. As a result the electric field in the plasma is altered and the discharge becomes more resistive.¹² The transition between the two regimes is generally believed to be determined by the residence time of the precursor gases in the reaction.

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chamber. In this article we will present data that suggest that the \( \alpha \rightarrow \gamma \) transition is not controlled by the residence time of the precursors in the reactor but by the gas composition and the pressure, in particular the partial pressure of silane and germane. The results presented in this article further show a strong correlation between the gas composition and the rate of powder formation in the plasma. We believe that the conclusions have serious implications for the design of large PECVD deposition systems for industrial processing.

The remainder of this article is organized as follows. In Sec. II we describe the experimental setups and procedures used for this research. We will show how the powder formation in the plasma is detected. The results of experiments are presented in Sec. III, and discussed in Sec. IV.

II. EXPERIMENT

The experiments were carried out in a two-chamber deposition system that is normally used for the deposition of amorphous silicon layers and solar cells for research purposes. High-quality material can be made in this system which is supported by the fact that single junction \( a \)-Si:H test solar cells have an initial energy conversion efficiency of more than 10%. Samples enter the system via a load lock, thereby allowing the reaction chambers to maintain a high vacuum. The reaction chamber used for our experiments has a background pressure of less than \( 10^{-7} \) mbar.

In the reaction chamber a glow-discharge plasma is created between two capacitively coupled electrodes. On the grounded top electrode, a glass substrate was mounted during the experiments. The interelectrode distance is 12 mm and the area of the powered electrode is 144 cm\(^2\). A 13.56 MHz rf signal is provided by a Manitou rf generator with an automatic matching network. For our experiments we used a power of typically 3 W. In some cases the automatic matching network was switched off in order to investigate its influence, but this did not affect the results. The pressure in the chamber during processing is measured by an MKS 127 baratron, connected to an MKS 600 unit that controls an MKS 625B butterfly valve. Each process gas passes through an MKS mass flow controller that in turn is controlled by an MKS 127 baratron, connected to an MKS 600 unit that controls an MKS 625B butterfly valve. Each process gas passes through an MKS mass flow controller that in turn is controlled by an MKS multichannel gas controller. The gases used in our experiments are SiH\(_4\), GeH\(_4\), and H\(_2\) with maximum flows of 50, 5, and 200 sccm, respectively. With these flows we can realistically achieve dilution ratios up to \( [\text{H}_2]/([\text{SiH}_4]+[\text{GeH}_4])=40 \).

The plasma was characterized by measuring the dc self-bias, \( V_{sb} \), over the electrodes. The self-bias is the difference of the potential drops over the plasma sheaths near the two electrodes and arises only if the electrode configuration is not symmetrical. The self-bias depends on the ratio of the grounded and rf electrode areas and is negative if the grounded electrode area is larger than that of the rf electrode. In practice the reaction chamber is not symmetrical, because the walls of the chamber will effectively contribute to the area of the grounded electrode and hence a self-bias will develop. A variation in the process pressure, the gas flow, or the gas composition will immediately result in a change of the self-bias. The self-bias is only an indirect characteristic of the plasma and by itself cannot give information about for instance, the plasma potential. Still, a variation of the self-bias indicates a change in the plasma physics, provided the other deposition parameters are kept constant. In this article we characterize the plasma by the peak-to-peak voltage of the oscillating self-bias, \( \Delta V_{sb} \). In Fig. 1, an example of the time variation of the self-bias is shown for a H\(_2\)-diluted SiH\(_4\)–GeH\(_4\) plasma at a pressure \( p=0.80 \) mbar and total gas flow \( \phi_{total}=20 \) sccm. The partial gas flows are \( \phi_{\text{SiH}_4}=8 \) sccm, \( \phi_{\text{GeH}_4}=2 \) sccm, and \( \phi_{\text{H}_2}=10 \) sccm. When keeping the pressure and the total gas flow constant, the self-bias varies remarkably periodically in time. In this particular case \( \Delta V_{sb}=8.11 \) V and the oscillation time is 42 s. It has been shown that these oscillations in the self-bias are correlated to powder formation,\(^10\) and the occurrence of these oscillations can be used to detect the onset of powder formation, the \( \alpha \rightarrow \gamma \) transition. The oscillations of the self-bias are believed to result from a spatial variation of the powder-forming region in the plasma. In this powder-forming region the particles “sweep up” the electrons and become negatively charged, thereby affecting the self-bias. The region in which the powder is formed expands until it reaches a critical size at which all the particles that were initially trapped in the region are pumped away and the process is repeated. An alternative explanation is that powder particles grow in the bulk of the plasma, sweeping up the electrons in the process leading to a lower electron density. At some stage the particle size reaches a critical value at which moment the neutral drag force of the gas expels the particles and the process is repeated.\(^11\) In both cases the oscillation time is a good measure for the rate of powder formation as this is related to the expansion rate of the powder-forming region or, if the explanation of Ref. 11 is adopted, the time required to reach the critical particle size. In this article we will base the discussion of the results using the first explanation of the oscillating self-bias given in Ref. 10. In Sec. III we will show results...
of $\Delta V_{sb}$ and the oscillation time as a function of the pressure.

III. RESULTS

In order to investigate the powder formation behavior of SiH$_4$–GeH$_4$ plasmas, we measured $\Delta V_{sb}$ as a function of the deposition pressure for several gas flow settings. In Fig. 2 results are presented for the same gas flow settings as in Fig. 1. In this figure three different pressure regimes can be clearly distinguished:

(i) Low-pressure range in which $\Delta V_{sb}$ is small and the plasma is stable. In this pressure range there is no powder formation in the plasma.

(ii) Medium-pressure range in which $\Delta V_{sb}$ varies significantly and the plasma is considered to be in the $\gamma'$ regime. The self-bias is clearly oscillating due to a spatial varying powder forming region.

(iii) High-pressure range in which $\Delta V_{sb}$ is again small, but the plasma is visually highly unstable.

This behavior is observed for all investigated gas compositions. For the sake of clarity we define the transition pressure from the low- to medium-pressure range as the pressure at which $\Delta V_{sb}$ is a maximum. Notice, however, that the oscillations start to occur at lower pressures and so does the powder formation. The oscillations in the self-bias suggest that the material properties of the growing film, in particular the Ge concentration, might oscillate as a function of depth. We have investigated this by carrying out a SIMS measurement on a layer that was deposited under severe oscillating self-bias conditions. In this layer one oscillation period corresponds to a thickness of about 23 nm, which is well within the detection limit of SIMS. We found that the oscillations in the self-bias were not reflected in the Ge/Si profile, although this does not imply that other material properties do not vary as a function of depth.

The effect of varying the total gas flow while keeping the gas flow ratios constant is shown in Fig. 3(a). With the exception of the smallest total gas flow (i.e., 10 sccm), the maximum $\Delta V_{sb}$ at this particular gas composition is found at a pressure of 0.80 mbar. The variation of $\Delta V_{sb}$ with $p$ is hardly influenced by the total gas flow, $\phi_{\text{total}}$, indicating that in this case the powder forming process is not dependent on the residence time of the gas but only on the pressure. The influence of the gas composition at a constant total gas flow is shown in Fig. 3(b). The gas composition appears to have a clear influence on the transition pressure. The transition from pressure range (i) to range (ii) occurs at lower pressures if the GeH$_4$ fraction in the source gas is increased. Having in mind that the total gas flow is not varied and that at a particular pressure the residence time of the gas in the reactor is constant, this implies that the chemical nature of GeH$_4$ is responsible for the variation of the transition pressure. It is known that the dissociation rate of GeH$_4$ is much higher than that of SiH$_4$ and it seems reasonable to assume that the explanation of the gas composition dependence of the transition pressure is related to this fact.

We have also investigated the effect of H$_2$ dilution on the powder formation. Hydrogen dilution is usually applied to obtain device quality $a$-Si$_{1-x}$Ge$_x$:H for application in solar cell devices. Figure 3(c) shows the results of experiments in which the H$_2$ gas flow was varied. Clearly the transition pressure shifts to higher values with increasing H$_2$ dilution. However, by increasing the H$_2$ dilution we have also changed the total gas flow in our experiment and thus the residence time of the source gas. In order to separate the

![Fig. 2. Typical variation of $\Delta V_{sb}$ with the deposition pressure. The three stages are described in detail.](image)

![Fig. 3. Variation of $\Delta V_{sb}$ as a function of the deposition pressure for several gas flow settings: (a) varying the total gas flow at the same gas composition; (b) varying the $[\text{GeH}_4]/[\text{SiH}_4]$ ratio at a constant H$_2$ dilution and total gas flow; and (c) varying the H$_2$ dilution at a constant $[\text{GeH}_4]/[\text{SiH}_4]$ ratio. In the legend the flows are given in sccm for SiH$_4$/GeH$_4$/H$_2$.](image)
It is difficult to determine the total gas flow and the H₂ dilution, so we compare the data in Fig. 3(c) with those in Fig. 3(a). It appears that at a constant \([\text{GeH}_4]/[\text{SiH}_4]\) flow ratio and total gas flow the transition pressure increases with the H₂ dilution. Analyzing this in more detail by taking into account the difference in the pumping speeds of the different gases, we see that the transition occurs at the same partial pressure of GeH₄ plus SiH₄, irrespective of the different hydrogen dilutions that are used. If we replotted the data of Fig. 3(c) as a function of the residence time of the gas in the reaction chamber (not shown here), it appears that the transition to a powder forming plasma occurs at the same residence time. The transition in the undiluted case, however, occurs at a higher residence time. We therefore conclude that it is the partial pressure of GeH₄ plus SiH₄ that determines the transition. The H₂ dilution enhances the transition pressure, but this is due to the fact that by increasing the H₂ flow at a particular working pressure the partial pressure of GeH₄ plus SiH₄ is reduced. These findings imply that there is no observable chemical effect of H₂ dilution, which is surprising as hydrogen is one of the reaction products in the dissociation of SiH₄ and GeH₄. We believe that the lack of a chemical effect of hydrogen is due to the high dissociation probability of GeH₄, which therefore consumes a high fraction of the energy that is dissipated in the plasma. As a result, less energy is available for H₂ dissociation. In conclusion, the combined results from the experiments carried out so far suggest that it is the gas composition and the partial pressure of GeH₄ plus SiH₄ rather than the total gas flow that determines the transition pressure. An enhancement of the hydrogen fraction increases the transition pressure whereas an increase in the germane fraction results in a reduction.

So far we have showed the dependence of \(\Delta V_{\text{ab}}\) on the pressure. The relation between the oscillation time and the deposition pressure for several gas compositions is presented in Fig. 4. The oscillation time increases when the deposition pressure is reduced, which is due to the fact that at lower pressures the rate of powder formation is slower and therefore the expansion rate of the powder-forming region is slower. From Fig. 4(a) we conclude that the relation between the oscillation time and the pressure does not depend on the total gas flow. Furthermore, we observe below \(P = 0.75\) mbar a steep increase in the oscillation time, which indicates a slower powder formation rate. Variation of the \([\text{GeH}_4]/[\text{SiH}_4]\) flow ratio has a large effect on the relation between the oscillation time and the deposition pressure as shown in Fig. 4(b). An increase of this ratio results in a reduction of the oscillation time and of the transition pressure as observed in Fig. 3(b) (note that the hydrogen dilution is constant). This means that an increased GeH₄ fraction in the source gas enhances the rate of powder formation, which is consistent with a higher decomposition rate of GeH₄. Changing the H₂ dilution while keeping the \([\text{GeH}_4]/[\text{SiH}_4]\) flow ratio constant results in a shift of the transition pressure [see Fig. 3(c)], but the oscillation time seems to follow the same relationship as in Fig. 4(a) as can be inferred from a careful comparison of Figs. 4(a) and 4(c). In Fig. 5 we have plotted the data of Figs. 4(a) and 4(c) together and clearly a unique relationship between the oscillation time and the pressure becomes evident, provided the \([\text{GeH}_4]/[\text{SiH}_4]\) flow ratio is constant. As we have shown above, the H₂ dilution shifts the transition pressure to higher values, though this seems to be accompanied by shorter oscillation times. Apparently, at the \(\alpha-\gamma'\) transition the powder formation rate is increased.

Fig. 4. Variation of the oscillation time as a function of the deposition pressure for several gas flow settings: (a) varying the total gas flow at the same gas composition; (b) varying the \([\text{GeH}_4]/[\text{SiH}_4]\) ratio at a constant H₂ dilution and total gas flow; and (c) varying the H₂ dilution at a constant \([\text{GeH}_4]/[\text{SiH}_4]\) ratio. In the legend the flows are given in sccm for \(\text{SiH}_4/\text{GeH}_4/\text{H}_2\).

Fig. 5. The oscillation time as a function of the deposition pressure at several gas flow settings, however, all with the same \([\text{GeH}_4]/[\text{SiH}_4]\) ratio.
when H₂ is added to the gas mixture, irrespective of the fact that the partial pressure of GeH₄ plus SiH₄ is not changed.

In Sec. IV we will discuss the results shown here from the viewpoint of the plasma chemistry and based on the work of Doyle et al.¹² Therefore we will focus on the reaction products that are believed to be involved in the powder formation. The effect of H₂ dilution can also be explained on the basis the plasma chemistry. First, however, we will pay some attention to the pressure dependence of the oscillation time.

### IV. DISCUSSION

As shown in the previous section the oscillation time becomes shorter with increasing pressure, indicating a faster powder formation rate, provided the critical size of the powder-forming region remains unchanged. We believe that at higher pressures the powder formation rate is enhanced, simply because the density of the gas is higher. At a higher gas density particles grow quicker, because the collision rate with other particles and radicals in the plasma is higher and as a result the critical size of the powder-forming region is reached faster.

As the basis for the discussion about particle formation we start with the main reactions that are found to occur in silane and germane plasmas,¹² respectively,

\[ \text{SiH}_4 \rightarrow \text{SiH}_3 + \text{H} \quad (<0.1), \]

\[ \text{SiH}_3 + 2\text{H} \quad (0.80–0.90), \]  

\[ \text{SiH} + \text{H} + \text{H}_2 \quad (0–0.2), \]

\[ \text{GeH}_4 \rightarrow \text{GeH}_3 + \text{H} \quad (0.80–0.85), \]

\[ \text{GeH}_3 + 2\text{H} \quad (0.15–0.20). \]  

(R1)

(R2)

Between the brackets the branching fractions of the dissociation reactions are given according to Ref. 12. It is generally accepted that SiH₃ and GeH₃ are responsible for the film deposition of amorphous silicon or germanium, respectively. The SiH₃ is mainly indirectly produced by the reaction

\[ \text{H} + \text{SiH}_4 \rightarrow \text{H}_2 + \text{SiH}_3, \]  

(R3)

thereby consuming H that is produced by reaction (R1). The SiH₂ that is formed in reaction (R1) reacts for a large part with SiH₄ to form Si₂H₆. In contrast to the formation of SiH₃, GeH₃ is to a large extent formed directly by reaction (R2) in addition to a similar reaction as (R3),

\[ \text{H} + \text{GeH}_4 \rightarrow \text{H}_2 + \text{GeH}_3. \]  

(R4)

Similar, to SiH₃, GeH₂ is held responsible for higher germane and silylgermane production.

For the deposition of α-SiGe:H out of a SiH₄ rich plasma the situation is more complicated. In that case the production of Si₂H₆ is suppressed, because SiH₂ preferentially reacts with GeH₄. Also the silane gas efficiency is enhanced as is reflected in a higher deposition rate and, according to Ref. 12, results from SiH₃ reacting with GeH₄. Furthermore, the results found by Doyle et al.¹² showed that deposition is mainly governed by reactions at the growing surface involving SiH₃ and GeH₃. They also reported a significant additional contribution to the growth of H₂SiGeH₂. A detailed discussion of the plasma chemistry of SiH₄–GeH₄ plasmas is beyond the scope of this article and is referred to in Ref. 12.

In this article we focus on the production of GeH₂. In a recent paper¹⁵ the high reactivity in the gas phase of this radical was revealed in GeH₂ rf discharges. It was also suggested that this radical is responsible for initial particle growth, because a similarity was observed in the spatial distribution in the plasma of the GeH₂ and particle density.¹⁴ In a GeH₄ plasma the initial particle growth is followed by a rapid growth phase in which particles coagulate at a high rate. We argue that a similar mechanism is responsible for powder formation in the SiH₄–GeH₄ plasmas discussed here. In this case GeH₂ is mainly produced by reaction (R2). The abundance of this radical in the plasma is of course dependent on the GeH₄ fraction in the gas phase. The higher the [GeH₄]/[SiH₄] flow ratio, the higher the GeH₂ production and thus the faster particles are formed. In pure SiH₄ plasmas the powder formation is generally related to the SiH₂ production. However, as indicated above the addition of GeH₄ to the precursor gas increases the gas efficiency, because the SiH₂ reacts with GeH₄. This implies that SiH₂ does not play a major role in the powder formation in SiH₄–GeH₄ plasmas, although we do not have evidence for this.

As described in the previous section, our results show that the variation of the transition pressure is dependent on the partial pressure of GeH₄ plus SiH₄ and not directly on the total gas flow, even if hydrogen is added to the gas mixture. However, by increasing the germane fraction not only a shift of the transition pressure is observed, but also the relation between the oscillation time and pressure is affected. Applying hydrogen dilution does not affect this relationship. It has been shown that hydrogen dilution shifts the α–γ transition pressure to higher values.¹⁵ However, when examining the results of the growth rate versus residence time, using the partial pressure of SiH₄, a common dependence of the data in the α regime and a common residence time value for the α–γ transition was observed. It should be noted that in the experiments in Ref. 15 the SiH₄ flow was not varied and hence also the residence time. In our experiments we have changed the total gas flow as well as the GeH₄ plus SiH₄ flow [Fig. 3(a)] and we observe no change in the transition pressure. Therefore we believe that it is the partial pressure of GeH₄ plus SiH₄ rather then the residence time that determines the α–γ transition. Strong H₂ dilution has been shown to be beneficial for the material quality and it has been suggested that hydrogen tends to etch preferably weak bonds on the growing film.¹⁶ The plasma chemistry can be changed also, as the addition of H₂ to the plasma enhances the production of atomic H, thereby pushing the reactions (R3) and (R4) forward resulting in a higher production of SiH₃ and GeH₃. The H₂ addition does not, however, affect the α–γ transition because the transition is dependent on the GeH₃ production which in turn is determined by the partial pressure of GeH₄ as described above. It can be argued that the GeH₃ production is affected when hydrogen is added to the gas mixture, as reaction (R4) consumes GeH₄. As a re-
sult of this reaction the powder formation rate should slow down, in contrast to the observations reported here. To explain the increase of the powder formation rate either another mechanism is required that balances the decrease of this rate due to the reduction of the GeH₂ production, or a mechanism in which the GeH₂ production is not affected. We argue that the production of GeH₂ is not affected, because GeH₄ dissociates much faster than SiH₄ and H₂. Consequently, a large fraction of the energy that is dissipated in the plasma is used for the GeH₄ dissociation and thus less energy is available for the dissociation of SiH₄ and H₂. The increase of the powder formation rate then might result from reactions of SiH₄ with the highly reactive GeH₂. With computer modeling it is possible that more insight can be obtained in the matter.

V. CONCLUSIONS

We have studied powder formation in SiH₄–GeH₄ plasmas by measuring oscillations in the self-bias. The oscillations in the self-bias are due to the spatial oscillation of the powder-forming region in the plasma and the onset of the oscillations, observed by monitoring the self-bias as a function of the deposition pressure, are believed to be related to the α–γ' transition. In addition, the oscillation time is an indication of the rate of powder formation in this region. Measuring the self-bias thus provides an easy tool to study particular plasma processes.

Our results show that the powder formation is determined by the [GeH₄]/[SiH₄] flow ratio. When this ratio is increased, the transition pressure at which powder formation starts to occur is reduced and the rate of powder formation is enhanced. We attribute the powder formation mainly to GeH₂ radicals in the rf discharge, which are very reactive and therefore excellent candidates for particle formation. Diluting the plasma with H₂ leads to an enhancement of the transition pressure. However, the results suggest that the transition pressure under hydrogen dilution conditions is controlled by the partial pressure of SiH₄ plus GeH₄ rather than by the residence time of the gas.

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