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Evaluation of the Electrical Asymmetry Effect by spectroscopic measurements of capacitively coupled discharges and silicon thin film depositions

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A B S T R A C T

A capacitively coupled hydrogen diluted silane discharge operated at pressures above 500 Pa and driven by a combination of 13.56 MHz and 27.12 MHz is investigated by infrared laser absorption spectroscopy. Adjusting the phase angle between the two applied frequencies enables the control of the discharge symmetry via the Electrical Asymmetry Effect (EAE). The absorption measurements prove that the degree of dissociation of SiH_4 and, thus, the radical flux remains approximately constant, independently of the phase angle. Furthermore, the results show that the DC self-bias can be adjusted by tuning the phase, so that the sheath voltages change. Thus, the ion properties at the electrodes can be controlled via the EAE. This has an effect on the deposition of thin films, which are grown on glass substrates at various phase angles and analyzed using Raman spectrometry: Reducing the ion energy at the substrate leads to a decrease of the amorphous fraction in the predominantly microcrystalline film.

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1. Introduction

In the manufacturing of various high-tech products such as glass coatings or thin film solar cells, the deposition of thin films on large area substrates is required. Here, plasma enhanced chemical vapor deposition (PECVD) is a common method, because the energy of the radicals and ions produced in the chemically rich gas phase can be controlled, thereby reducing the harmful thermal impact on sensitive materials [1]. In particular, capacitively coupled radio frequency (CCRF) discharges are widely used, as this discharge type allows for a very uniform treatment of plain surfaces [2]. Both the general quality and the specific physical properties of the thin film are mainly determined by the properties of the chemically reactive radicals and ions reaching the layer growing on the substrate surface. Hence, methods of controlling the flux ratio of the ions and radicals as well as methods of controlling the total fluxes and incident energies of radicals and ions are highly desired.

Recently, many studies demonstrated that a collisional discharge regime, i.e. the operation of the CCRF discharge at relatively high pressures of several hundred Pascal, is beneficial for the deposition of thin silicon films [3–6]. This can be attributed to the fact that the ion energy is strongly reduced due to the ion mean free path being much shorter than the width of the sheath region, where the ions are accelerated towards the surface by the rf oscillating electric field. Thus, damaging of the growing film by highly energetic ion bombardment is avoided. However, in general any change in global control parameters such as the applied voltage amplitude or the gas pressure affects both the energy and the flux of ions reaching the electrodes. Hence, there is no method of separate control of the flux and mean energy of the ions arriving at the electrodes in capacitive single-frequency plasmas [7].

The Electrical Asymmetry Effect (EAE) [3,4,8–14] provides a simple means of independent control of ion energy and ion flux by applying a dual-frequency voltage waveform

$$\phi_{\sim}(t) = \phi_{lf}\cos(\omega t + \theta) + \phi_{hf}\cos(2\omega t) \quad (1)$$

to the powered electrode. ϕ_{lf} and ϕ_{hf} are the amplitudes of the fundamental angular frequency (ω) and its second harmonic (2ω), respectively. Adjusting the phase angle, θ, between the two frequencies allows for

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the control of the symmetry of the voltage waveform. Accordingly, the global extrema of $\phi_{\sim}(t)$ and, thereby, the DC self-bias developing in the CCRF discharge can be controlled by tuning θ [8–13]. This affects the sheath voltages, which, in turn, are most important for the ion energy at the electrodes. Therefore, the symmetry control via the EAE results in a convenient control of the mean ion energy independently of the ion flux [8,9,11,14].

A very similar approach called voltage waveform tailoring (VWT) [15–22] is based on the application of multiple synchronized frequencies to the capacitively coupled plasma, thereby enhancing the control range [15,23,24]. Johnson et al. have demonstrated that this effect is indeed useful for the optimization of CCRF discharges for thin film deposition applications [19–21]. However, this approach is associated with practical problems, as the matching of more than two subsequent frequencies is technically challenging and has not been achieved yet. Nevertheless, these studies show that the control of the voltage waveform translates into a control of the film morphology in PECVD applications [22]. Fundamentally even more important, gaining insight into the specific role of the fluxes and energies of radicals and ions might be possible using the EAE or VWT, respectively.

The focus of this paper is to emphasize the decoupling between the radical fluxes and the ion properties using the EAE at high pressures and to show how the ion energy control via the EAE affects the properties of microcrystalline silicon thin film depositions. The experimental setup and, in particular, the spectroscopic methods used to investigate the plasma and the deposited films, respectively, are explained in the next section. The results are presented and discussed in section three. Finally, we summarize our findings in the fourth section.

2. Experimental setup

The basic setup of the CCRF parallel plate discharge used in this work has been described in detail elsewhere [12–14]. In brief, a plasma is operated in the 14 mm gap between the quadratic powered electrode with a surface area of 1600 cm^2 and the grounded counterelectrode. The temperature of both electrodes can be set individually and is maintained constant. The feed gas consisting of H_2 with a small admixture of SiH_4 flows into the chamber through the showerhead system implemented into the powered electrode. A voltage waveform according to Eq. (1) is produced by two synchronized rf generators. Each of the two frequencies has its own matching branch, and is blocked from the other matching branch by a low or high pass filter, respectively. The amplitudes of both frequencies, the phase angle, and the DC self-bias are determined from high voltage probe measurements after a calibration procedure [9].

The dissociation of the SiH_4 feed gas is measured by infrared laser absorption spectroscopy (IRLAS, see Fig. 1). By sweeping the current, a lead salt laser diode, which is kept at a constant temperature within a liquid nitrogen dewar, is tuned in the spectral range around 2230 cm^{-1} (or 4484 nm) corresponding to the R branch of the ν_3 vibration band of the SiH_4 molecule. A monochromator with a grating blazed at 4750 nm is used to select one of the multiple modes, which the diode emits simultaneously. The overall laser mode light intensity is then split into three components. A fraction of 25% is detected behind a reference cell filled with N_2O gas to find absolute spectral positions [25], while a fraction of 25% passes an etalon to visualize relative spectral changes. The analysis of both signals, which are measured by capacitively coupled HgCdTe detectors, allows for the determination of the absolute wavelength scale. The remaining 50% of the beam is coupled into the discharge chamber.

After a double path through the plasma chamber, the laser signal is measured by a directly coupled detector. The length of the laser beam path within the H_2/SiH_4 chamber is about 2.83 cm = 166 cm. Accordingly, the region of a homogeneous plasma adjacent to the powered electrode with an edge length of 40 cm, where dissociation takes place, corresponds to approximately 50% of the total absorption length. From these measurements, the absorbance, A', can be obtained as

$$A' = -\ln\left(\frac{I}{I_0}\right). \quad (2)$$

Here, I and I_0 are the intensities in the presence and absence of absorption, respectively. Four absorption lines are investigated, which are spectrally located at 2230.211 cm^{-1}, 2230.249 cm^{-1}, 2230.338 cm^{-1}, and 2230.374 cm^{-1}, respectively. These lines can be assigned to transitions from $J = 8$ states to $J = 7$ states of the $^{30}SiH_4$ isotope according to the outcome of simulations [26]. The absorbance is directly proportional to the density of the absorbing medium. Hence, in the following the reduction of the absorbance is used as a measure for the reduction in the SiH_4 density, i.e. for the increase in the dissociation of the SiH_4 feed gas molecule.

Thin film depositions, which have been grown on 10 × 10 cm^2 glass substrates placed centrally on top of the grounded electrode, are analyzed by means of Raman spectrometry. A HeNe laser emitting at 632.8 nm is focused on the film by a microscope lens. It has been assured that the incident laser power does not cause any irreversible damage, e.g. phase changes, to the deposited film [27]. The Raman shift, i.e. the difference in the wavenumbers of the incident and scattered light, has been calibrated by the examination of a monocrystalline silicon probe. In addition, a *Dektak* profilometer is used to estimate the film thickness and deposition rate, respectively, by moving the needle across a film edge, which is produced by a clamp that covers a small part of the glass substrate during the deposition process. Other diagnostic techniques such as Fourier transform infrared (FTIR) spectroscopy [28,29] would further improve the film characterization. However, such

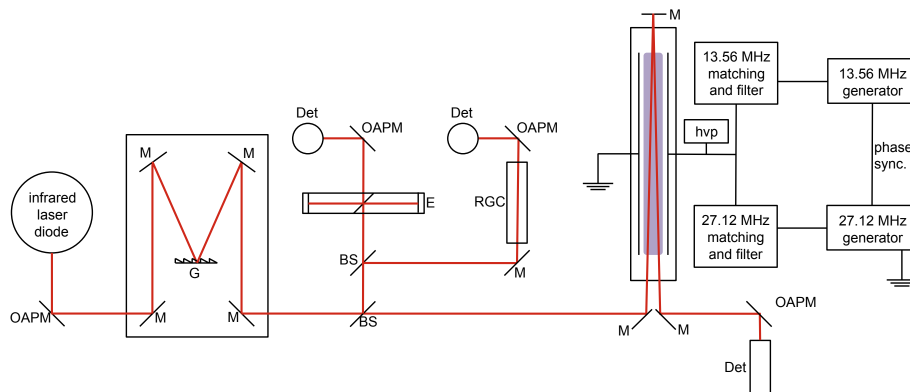

Fig. 1. Sketch of the IRLAS system and the discharge setup. OAPM: off-axis parabolic mirror, M: mirror, G: grating, BS: beam splitter, E: etalon, RGC: reference gas cell, Det: detector, hvp: high voltage probe.

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