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A capacitive probe with shaped probe bias for ion flux measurements in depositing plasmas

M. C. Petcu,[1,a)] A. C. Bronneberg,[1] A. Sarkar,[1] M. A. Blauw,[2] M. Creatore,[1] and M. C. M. van de Sanden[1]

[1]*Department of Applied Physics, Eindhoven University of Technology, P.O. Box 513, 5600 MB Eindhoven, The Netherlands*
[2]*Holst Centre, High Tech Campus 31, 5656 AE Eindhoven, The Netherlands*

The application of a pulse shaped biasing method implemented to a capacitive probe is described. This approach delivers an accurate and simple way to determine ion fluxes in diverse plasma mixtures. To prove the reliability of the method, the ion probe was used in a different configuration, namely, a planar Langmuir probe. In this configuration, the ion current was directly determined from the *I-V* characteristic and compared with the ion current measured with the pulse shaped ion probe. The results from both measurements are in excellent agreement. It is demonstrated that the capacitive probe is able to perform spatially resolved ion flux measurements under high deposition rate conditions (2–20 nm/s) in a remote expanding thermal plasma in $Ar/NH_3/SiH_4$ mixture. © *2008 American Institute of Physics.* [DOI: 10.1063/1.3020709]

I. INTRODUCTION

Nowadays the use of plasmas is widespread in many industrial applications, e.g., solar cells, microelectronics, and biomaterials. The understanding of a plasma system is a very important issue for any of the applications mentioned above, especially in the area of thin film processing (etching and/or deposition), where ions and their interaction with surfaces are found to play a fundamental role. The industrial demands for fast and advanced detection require plasma diagnostic tools to control and monitor the plasma processes. Several techniques to determine ion fluxes under different plasma conditions have been developed.[1–5] Langmuir probe is one of the most widely used diagnostic techniques to measure ion fluxes in a nondepositing plasma system.[6,7] However, the measurement of the ion fluxes in depositing plasmas is still a challenge since insulating layers deposited on the probe surface can seriously limit the Langmuir probe accuracy. In order to overcome this problem, several authors have proposed an *in situ* cleaning procedure by biasing the probe to high voltages[8,9] (up to 100 V) or by including film growth in the probe analysis.[10]

An alternative method to measure the ion flux has been recently proposed by Braithwaite *et al.*[11] By using a short periodically chopped rf pulse ($\sim 100~\mu$s) to bias a planar probe, the current and voltage are monitored during the discharging of an external capacitor connected in series with a large single side disk guarded by a ring. This approach is particularly suitable to measure ion fluxes in processing plasmas as long as the thickness of the deposited layer on the probe surface is below a certain critical value. The charging behavior of the external capacitor is linear in the high bias portion of the characteristic and the ion flux can be calcu-

lated by a linear fit to this region. Since the probe is discharging during the phase, the sheath properties vary, i.e., the voltage over the sheath decreases. The technique was demonstrated for a reactive ion etching setup using an Ar/CF_4 plasma mixture, where the probe will be coated with a layer due to the plasma deposition process itself as well as from residues originating from the reactor walls.

In this contribution a biasing method based on the use of a pulse shaped waveform signal has been applied to a capacitive probe. The use of a pulse shaped waveform was introduced by Wang and Wendt[12] in order to control the energy distribution of the ions impinging on the substrate during plasma processing. Instead of using a sinusoidal waveform as proposed by Braithwaite *et al.*,[11] a periodic bias voltage has been chosen for our ion probe experiments. The shape of the waveform signal is characterized by a short voltage pulse in combination with a linear and longer voltage ramp. The short pulses are needed in order to prevent charge accumulation on the probe surface. The linear voltage ramp of the signal, associated with the charging of the external capacitor connected in series with the collecting surface of the probe, enables an easy ion flux calculation, as we will show below.

A possible advantage of our approach compared with the one proposed in Ref. 11 is that measurements are made with the sheath voltage held constant at high negative bias. This is maintained by drawing the steady ion current into an external capacitor that therefore charges at a rate directly proportional to the ion current. To achieve this condition the voltage ramp rate is adjusted in the measurement procedure. As a result, using this waveform signal gives an easier and more accurate way to obtain the ion flux from the discharging rate of the capacitor. Such an approach could be easily implemented in a sensor for ion flux control in various discharges, e.g., in the case of a biased substrate to induce ion bombardment during film growth or etching for different applications.

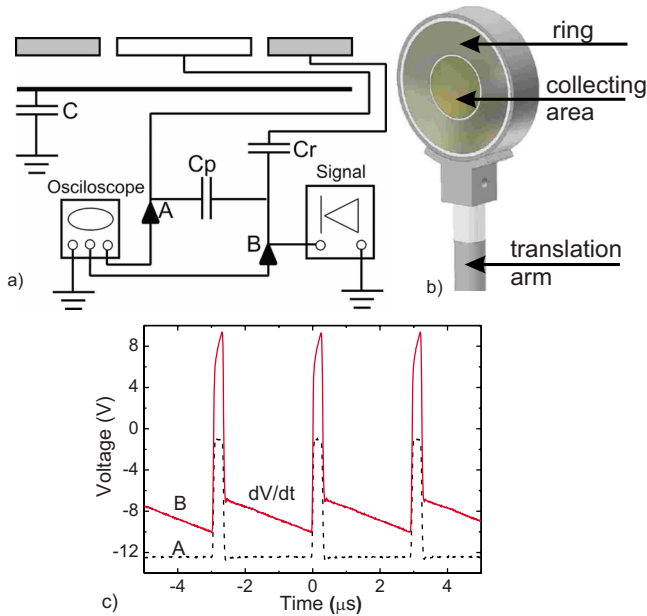a)Electronic mail: m.c.petcu@tue.nl.

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FIG. 1. (Color online) (a) The electrical circuit of the ion probe. (b) Three-dimensional picture of the ion probe. (c) The two measured voltages: the probe voltage (A, dashed line) and the voltage generator output (B, solid line).

II. EXPERIMENTAL

A schematic of the capacitive probe (hereafter referred to as ion probe) used in this work is presented in Fig. 1(a). The collecting area (inner surface) is connected in series with a capacitor C_p (typically 1.5 nF). In order to reduce the edge effects that may occur due to the potential drop in the probe sheath region, a ring (outer surface) connected in series to a capacitor C_r (typically 5 nF) is used. Both inner and outer surfaces are electrically separated by a ceramic dielectric and placed in a grounded cup [see Fig. 1(b)]. The values of the capacitors are chosen to satisfy the following equation:

$$A_{inner}/A_{outer} = C_p/C_r, \tag{1}$$

where A_{inner} and A_{outer} are the areas of the probe and ring, respectively. A bias voltage, V_{bias} (usually −10 V), is applied using a Hewlett Packard 32120 waveform generator. As mentioned before, the waveform signal is characterized by a short pulse and a ramp period with a ratio of approximately 0.25 [see Fig. 1(c)]. The measured voltages, i.e., the probe voltage (signal A) and the voltage generator output (signal B), are monitored by means of an oscilloscope [cf. Fig. 1(c)]. The ion flux discharges the capacitor C_p and the voltage is maintained constant by changing the slope of the applied bias signal via the frequency of the bias voltage signal, usually in the range of 1–300 kHz.

The ion flux experiments reported in this paper are performed in a depositing plasma with deposition rates in the range of 2–20 nm/s. The deposition technique utilized is the so-called remote expanding thermal plasma (ETP) already extensively described in literature,[13] so a brief summary suffices here. The plasma source is a cascaded arc with a 2.5 mm arc channel operating in Ar, having a flow rate of 33 sccs (standard cubic centimeter per second). The arc current is 45 A and the voltage is 70 V, operating at subatmospheric pres-

sure, typically 600 kPa. The plasma is generated between three cathodes and an anode plate and expands through a nozzle in the deposition chamber. NH_3 and SiH_4 are introduced downstream through two injection rings placed at 20 and 23 cm from the nozzle, respectively. The pressure in the downstream region is kept at 15 Pa during the measurements. The capacitive probe is placed at 15 cm from the nozzle. In this paper, all the ion flux measurements have been performed in Ar/NH_3 and $Ar/NH_3/SiH_4$ plasma mixtures, which are used, e.g., to deposit silicon nitride thin films for antireflection layers in solar cells.[14,15]

In order to check the reliability of the measurements performed with the capacitive probe, a comparison in non-depositing Ar/NH_3 plasma with a planar Langmuir probe is performed. The Langmuir probe measurements are carried out using the ion probe as a planar Langmuir probe. The collecting area of the probe was connected to a power supply by means of a Keithley 2400 source unit. By applying a voltage between −20 and 5 V, the current is collected resulting in a *I-V* characteristic.

The measurements for the comparison were performed at the central position of the reactor in identical plasma conditions (e.g., reactor pressure and distance from plasma source) of the ion probe work by varying the NH_3 flow rate with values between 0 and 10 sccs, which results in a variation in the ion flux by at least two orders of magnitude.[16,17] The ion current obtained from the *I-V* measurements corresponding to $V_{bias}=−10$, $I_{i,LP}$, was plotted as a function of the ion current $I_{i,IP}$, which resulted from the ion probe measurements. In the case of the ion probe measurements in a high density Ar plasma, the collecting area needed to be made smaller to perform accurate ion probe measurements. The adjustment of the probe design was done by placing a ceramic with a 2 mm diameter on top of the collecting area, enabling the measurement of ion fluxes over a range of $10^{15}–10^{20}$ cm^{-2} s^{-1}. All the measurements were performed with an accuracy of about 15%. This includes 13% standard deviation due to the reactor conditioning determined from the statistics over several measurements performed in the same conditions, 1% error from the data analysis (e.g., the ramp fitting procedure) and 1% measurement error (e.g., the instrument reading).

During a pulse period, the capacitor is alternately charged and discharged with electrons and ions, respectively. The charge Q_{C_p} collected by the external capacitor C_p can be calculated using the following equation:

$$Q_{C_p} = \int_0^t I dt, \tag{2}$$

where I is the current flowing through the probe circuit. In general, the probe bias can be determined using Eq. (3)

$$eA_{inner}(\Gamma_i - \Gamma_e) = C_p(dV/dt)_{bias}, \tag{3}$$

where Γ_i and Γ_e are the ion and electron fluxes reaching the probe surface, respectively, and $(dV/dt)_{bias}$ is the first derivative of the potential developed between the probe surface and ground.

Since we are interested only in the ion flux, the current measured at a constant high negative bias (for a bias $\gg -kT_e$) is totally determined by the ions, e.g., Eq. (3) becomes

$$eA_{\mathrm{inner}}\Gamma_i = C_p(dV/dt)_{\mathrm{measured}}, \tag{4}$$

where $(dV/dt)_{\mathrm{measured}}$ is the first derivative of the voltage drop over C_p and ground. This first derivative can be easily determined by linearly fitting the ramp of the output signal [signal B, Fig. 1(c)].

Due to high deposition rates specific to ETP, thick films (>100 nm) can be deposited on the probe surface in a relatively short time. Basically, this means that an additional capacitor due to the presence of the grown film on the probe surface needs to be included in the circuit [Fig. 1(a)]. The film grown induces a significant voltage drop across the layer, therefore, inducing a change in the ion current in the probe circuit. Nevertheless, a certain thickness d_{crit}, here defined as critical thickness, can be tolerated, below which still reliable results can be obtained. For thicker films $d > d_{\mathrm{crit}}$, the film influence becomes significant and needs to be taken into account. The critical film thickness, d_{crit} follows from the condition that the capacitance of the film deposited on the probe is much higher than C_p, i.e.,

$$d_{\mathrm{crit}} < \varepsilon_0 \varepsilon_r A_{\mathrm{inner}}/C_p, \tag{5}$$

where ε_0 is the vacuum permittivity and ε_r is the dielectric constant of the film (~ 4 for SiN_x thin films). Substituting these values, a critical film thickness of approximately 4 μm is obtained.

In order to verify the influence of the estimated critical film thickness on the measurements, an experimental procedure has been established. The measurements are performed radially from the central position to the reactor wall and for each point the ion current is measured three times. The first measurement in nondepositing Ar/NH_3 plasma is taken as a reference before SiH_4 injection. After the second measurement in $Ar/NH_3/SiH_4$ plasma, which generates SiN_x film,[14,15] a third measurement in Ar/NH_3 plasma is performed and compared with the reference measurement. The ion flux Γ_i is calculated according to Eq. (4). Although the results are not shown in this paper, the two ion fluxes before and after SiH_4 addition coincide within the experimental errors, confirming that at the given deposition rates the ion flux measurements are not influenced by the film presence as long as $d < d_{\mathrm{crit}}$.

III. RESULTS AND DISCUSSION

A comparison between the ion currents measured using the pulse shaped ion probe in two different configurations, namely, planar Langmuir probe and standard ion probe (pulse shaped probe configuration) in a nondepositing plasma, is shown in Fig. 2. The results show a very good agreement between the two configurations used, as confirmed by the fitted slope of 1 ± 0.03 (cf. Fig. 2). In addition, from the ion probe current measurements the ion flux was calculated, the results are represented in Fig. 3. The values
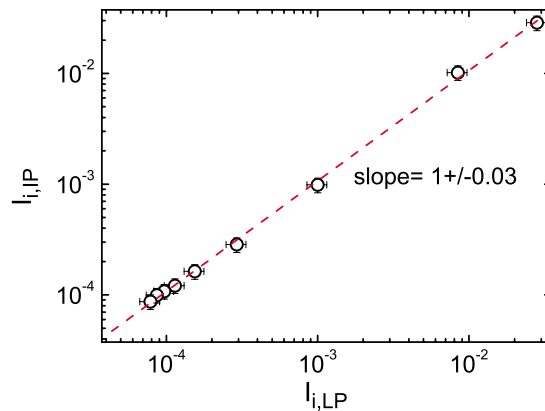

FIG. 2. (Color online) Comparison between the ion currents measured in Ar/NH_3 plasma mixtures with the ion probe used in standard configuration (pulse shaped bias) and the ion probe used in a planar Langmuir probe configuration reported at -10 V. The measurements were performed at 15 cm far from the plasma source at the central position of the reactor. Reactor pressure 15 Pa, Ar flow 33 sccs, NH_3 flow 0–16 sccs. The dashed line is a linear fit with an intercept through 0.

cover a wide range of fluxes from 10^{18} cm^{-2} s^{-1} in pure Ar plasma to 10^{15} cm^{-2} s^{-1} when a maximum of NH_3 is injected.

The results of the ion flux measurements in Ar/NH_3 and $Ar/NH_3/SiH_4$ plasma mixtures (for film thickness on the probe smaller than the critical film thickness) are shown in Fig. 4. The radial profiles are Gaussian-like, having a maximum at the central position of the reactor and decreasing toward the reactor walls. The decrease in the ion flux by NH_3 and SiH_4 addition is related to the development of the plasma chemistry on the basis of the argon ion consumption as governed by two important processes in ETP. These processes, namely, charge transfer reaction between the argon ions emanating from the plasma source and NH_3 and/or SiH_4 molecules, followed by dissociative recombination with low energy electrons, have been reported in detail elsewhere.[13,16,17] The ion flux is gradually decreasing from 10^{18} cm^{-2} s^{-1} in pure Ar plasma as measured with the probe to 10^{15} cm^{-2} s^{-1} when a maximum of 16 sccs of NH_3 is injected (cf. Fig. 4). Due to the substantial Ar ions consumption via the two plasma processes mentioned above, the addition of 1 sccs of SiH_4 does not further promote a decrease in ion flux. There-
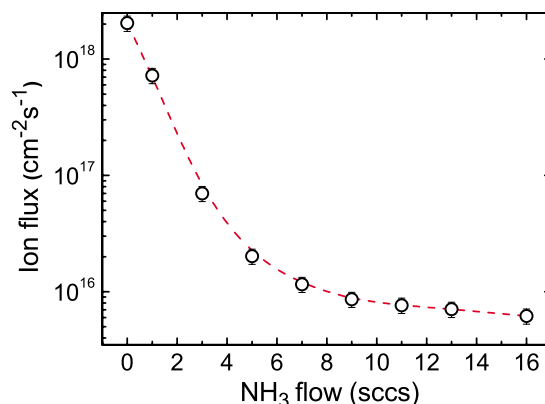

FIG. 3. (Color online) The ion flux resulted from the ion probe measurements at the central position of the reactor. Reactor pressure 15 Pa, Ar flow 33 sccs, NH_3 flow 0–16 sccs. The dashed line serves as a guide to the eyes.
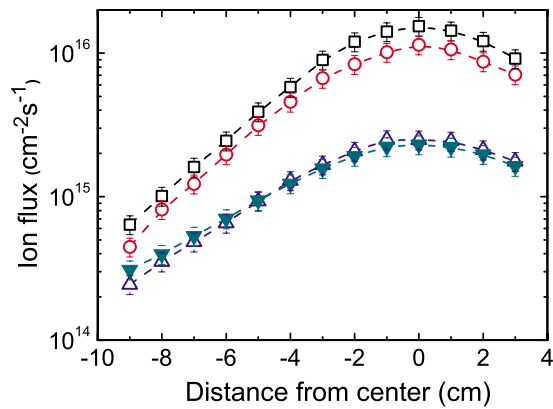
FIG. 4. (Color online) Spatially resolved ion flux measurements in Ar/NH$_3$ and Ar/NH$_3$/SiH$_4$ plasma mixtures: (□)—NH$_3$ flow 4 sccs, Ar flow 33 sccs, SiH$_4$ flow 0 sccs; (○)—NH$_3$ flow 6 sccs, Ar flow 33 sccs, SiH$_4$ flow 0 sccs; (△)—NH$_3$ flow 16 sccs, Ar flow 33 sccs, SiH$_4$ flow 0 sccs; (▼)—NH$_3$ flow 16 sccs, Ar flow 33 sccs, SiH$_4$ flow 1 sccs. The measurements are performed radially. The zero corresponds to the central position of the plasma chamber at 15 Pa. The dashed line serves as a guide to the eyes.

fore, above a NH$_3$ flow rate of 16 sccs, no further enhancement of the dissociation of SiH$_4$ molecules is achievable and the ion flux remains constant (cf. Fig. 4).

IV. SUMMARY

To summarize, an alternative version of the ion flux capacitive probe as proposed by Braithwaite *et al.*,[11] suitable to measure spatially resolved ion fluxes in depositing and nondepositing plasma mixtures, has been developed. This method was successfully implemented in a high rate deposition ETP system, showing the probe tolerance toward the presence of insulating layers. A comparison between the ion probe and the planar Langmuir probe shows an excellent agreement for fluxes in the range of 10^{15}–10^{20} cm^{-2} s^{-1}. The ion flux results in Ar/NH$_3$ and Ar/NH$_3$/SiH$_4$ plasma mixtures show a decrease with the addition of NH$_3$ and SiH$_4$ to the Ar plasma. Three orders of magnitude decrease in ion flux is observed in Ar/NH$_3$ plasma, i.e., from 10^{18} to 10^{15} cm^{-2} s^{-1}, when 16 sccs of NH$_3$ is injected. Moreover, in order to be able to perform ion flux measurements for a wide range of ion fluxes corresponding to different plasma mixtures, the design of the probe and/or the electrical circuit of the probe can be easily modified. The method used and described in this paper makes the ion probe a feasible tool that can be adapted to an industrial sensor in order to monitor the film growth for different applications or to control the ion flux in various plasma discharges.

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