Electrostatic probe measurement of low-pressure electronegative SF$_6$ discharges

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ABSTRACT

Electrostatic probe measurements for low-pressure inductively coupled SF$_6$ plasmas are performed. From the current–voltage (I–V) curves of the probe, the saturation currents of the positive ions, the electron density, and the electron temperature are measured. The electronegativity and the positive ion density are obtained by several different methods (the ion saturation current at the floating potential, the positive and negative current balances at the floating potential, and the three pressure point method). They are compared with the positive ion density obtained by the orbital-motion-limited theory.

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1. Introduction

Electronegative gases such as SF$_6$, oxygen, chlorine, and fluorocarbons are used extensively in discharges for various applications of plasma processing such as negative-ion assisted etching and charge-free ion implantation in semiconductor manufacture [1]. The presence of negative ions complicates the transport properties of the discharge. There has been considerable scientific and technological interest in electronegative plasmas [2–5] and there have been various methods in the determination of negative ion density [6–9]. Electrostatic probes [10–13], laser photodetachment (LPD) [6,7], and laser Thomson scattering (LTS) [14] are frequently-used diagnostic tools for detecting negative ions in plasma, among which electrostatic probe technique is simple and inexpensive, and provides the spatial resolution of plasma parameters.

The interpretation of current–voltage (I–V) characteristics of the electrostatic probe has been performed by different methods. If there is no negative ion, the electron density can be obtained either by measuring the electron saturation current or by integrating the electron energy distribution function (EEDF) integrals. If there are negative ions, it is proper to use the positive and negative ions for the electron density, if both ion densities are known since it is difficult to determine the plasma potential and the electron saturation current from the probe I–V curve. However, unless both the density and the temperature of the negative ion are very large comparing with the density and temperature of the electron, electron density could be calculated from the electron saturation current approximately due to huge mass ratio of the negative ions to electrons.

In order to deduce the density of negative ions from the probe I–V data, practical methods to compare the ratios of ion and electron saturation currents were introduced by Amemiya [15], Cooney et al. [16], and Shindo et al. [17]. Chabert et al. introduced a two-probe method to deduce the ratio of negative ion density to electron density [18,19]. However, these methods need many other information: temperatures of positive and negative ions, sheath potential, sheath area for positive ion collection, and effective mass of positive ions. In an earlier work [20], a simple probe method to determine the electronegativity was introduced and tested for inductively coupled SF$_6$ plasmas. That was called the three pressure point method and the electronegativity and the negative ion density were deduced by using the ratios of parameters such as the saturation currents of the positive ions and electrons, and the electron temperature at three adjacent pressure points. This method gives the positive ion density as a sum of the negative ion density and the electron density.

One of the concerns in electronegative plasmas is that how much is the difference between the actual positive ion saturation current and $I_{is} = 0.6eAn_+c_s$ (assuming that the positive ions have the normal Bohm speed $c_s = \sqrt{kT_e/M_+}$ at the sheath edge, A is the sheath area for positive ion collection, k is the Boltzmann constant, T_e is the electron temperature, and M_+ is the mass of positive ion) at the plasma potential. Since the measured ion current by cylindrical Langmuir probe does not saturate and the contribution from negative ions is difficult to assess, the application of $I_{is} = 0.6eAn_+c_s$ at the plasma potential is not easy.

In this work, the positive ion density is first obtained by the measured ion saturation current at the floating potential [21]. The positive ion density is also obtained by utilizing the positive and negative current balances at the floating potential [18]. They are then compared to those obtained by the three pressure point method and by the orbital-motion-limited theory. The accuracy of the three point

method is checked again by comparing with the results obtained by several different methods.

2. Theory

In electronegative plasmas, the electron density can be determined by

$$n_e = \frac{4I_{es}}{eA_p(8kT_e/\pi m_e)^{0.5}}, \tag{1}$$

where I_{es} is the electron saturation current, e is the electron charge, A_p is the probe area, and m_e is mass of electrons. This would hold if

$$\zeta \equiv I_{es}/I_{-s} = \left(0.25A_p e n_e v_e\right)/(0.6 A e n_- v_-) \tag{2}$$
$$= 0.4\left(A_p/A\right)(n_e/n_-)(M_-/m_e)^{0.5} \gg 1,$$

where I_{-s} is the saturation current of negative ions, n_- is the negative ion density, v_e is the average thermal speed of electrons, and M_- is the mass of negative ion. Here negative ions are assumed to be collected by the same way as the positive ions, i.e., they are passing the sheath with the normal Bohm speed (v_-). If negative ions are to be governed by the Boltzmann relation as electrons, ζ becomes $(n_e/n_-)(M_-/m_e)^{0.5}$, which is much larger than unity for most cases. As for the electron temperature, either the logarithmic slope of I–V curve or electron energy distribution function (EEDF) can be used by assuming the Boltzmann electrons or isotropic electron distribution. We observed that both methods yield almost the same values of the electron temperature, indicating one-temperature electrons and confirming the Boltzmann electrons for our experimental conditions [20]. Moreover, the electron density obtained by Eq. (1) compares well with that obtained by EEDF integral. This allows one to use Eq. (1) to find the electron density in electronegative plasmas.

As for the positive and negative ion densities, one can utilize the method of Ref. [18,19]. The positive ion saturation current in an electronegative plasma is described as

$$I_{is} = eA\Gamma_s(\alpha, \gamma_\pm, n_e), \tag{3}$$

where $\alpha = n_-/n_e$ is the density ratio of negative ions to electrons, $\gamma_\pm = T_e/T_\pm$. The temperatures of positive ions and negative ions are, T_+, and T_-, respectively. $\Gamma_s(\alpha, \gamma_\pm, n_e) = n_{+s}v_{+s}$ is the modified Bohm flux, n_{+s} and v_{+s} are the density and velocity of positive ions at sheath edge when negative ions are present.

The ratio of I_{is} to I_{es} is then written as

$$\frac{I_{is}}{I_{es}} = \sqrt{\frac{2\pi m_e}{M_+}}\frac{A}{A_p}\frac{\Gamma_s(\alpha, \gamma_\pm, n_e)}{n_e c_s} \tag{4}$$

In order to calculate the modified Bohm flux, one has to solve the fluid equation for positive ions combined with Poisson's equation for the electric potential. This model allow us to obtain the profiles of electric potential, positive ion flux, positive ion density in the presheath, and the I–V characteristic curve of the probe. In the numerical calculation of the positive ion flux, we consider that electronegative plasma consists of three charged species, and both the electron and negative ion densities obey Boltzmann relations. For a cylindrical geometry, the equations are one-dimensional assuming no orbital motion of the ion. The ions are all drawn radially into the probe. This theoretical model of the positive ion flux for planar [22], cylindrical [23,24] and spherical probes has been developed [25].

If a careful comparison of the measured ratio of I_{is}/I_{es} with the calculated Bohm flux is made, one can determine α and $\gamma_\pm$ in principle. Once α is evaluated, one can obtain the negative ion density

and this can be compared with the results obtained by the three point method.

The floating potential V_f is the potential at which the positive and negative currents to a probe balance, so that

$$\Gamma_s A_f = n_e A_p \sqrt{\frac{kT_e}{2\pi m_e}}e^{-e\Delta V/kT_e} + n_- A_p \sqrt{\frac{kT_-}{2\pi M_-}}e^{-e\Delta V/kT_-} \tag{5}$$

with $\Delta V = V_p - V_f$ and $A_f = A(V_f)$ are the sheath areas of a probe at the floating potential. The second term can be neglected under usual circumstances. If the approximations $\Gamma_s = 0.6 n_+ c_s$ and $n_+ = n_e + n_-$ are used,

$$0.6 n_+ A_f c_s = n_e A_p \sqrt{\frac{kT_e}{2\pi m_e}}e^{-e\Delta V/kT_e}. \tag{6}$$

The A_f can be estimated from either the Child–Langmuir law or the ABR theory [21]. With measured values of V_p, V_f, n_e, T_e, and effective ion mass M_+, n_+ can be calculated and the negative ion density is obtained.

The positive ion density can also be evaluated from the measured ion current at the floating potential ($I_i(V_f) = 0.6 e A_f n_+ c_s$). If we assume that the positive ion current I_i to cylindrical probes tend to follow an $I_i \propto (V_p - V_{pr})^{3/4}$ (Child–Langmuir law, V_{pr} is the probe voltage and V_p is the plasma potential), by extrapolating to the floating potential, one can obtain an estimate of the positive ion current at V_f. In order to know A_f, the sheath thickness should be determined. The sheath thickness was estimated from the Child–Langmuir law and the ABR theory at the floating potential [21].

To determine the positive ion density from another way, the orbital-motion-limited (OML) theory can be used for the low density plasmas with the thick sheath, and the ion positive current (I_i) is expressed as

$$I_i = n_+ e A_p \sqrt{\frac{kT_e}{2\pi M_+}}\frac{2}{\sqrt{\pi}}\left[\frac{e\left(V_p - V_{pr}\right)}{kT_e}\right]^{\frac{1}{2}}, \tag{7}$$

In the positive ion saturation region where the probe current is virtually all positive ion current, we can plot $I_i^2 = AV_{pr} + B$. From the slope, we can determine the positive ion density.

3. Experiment

In this study, an inductively coupled SF_6 RF plasma is employed as an example of electronegative discharge since SF_6 plasmas have found numerous applications in plasma processing such as dry etching of silicon. The plasma generation chamber consists of a stainless steel cylinder with a diameter of 28 cm and a length of 30 cm. A 1.9 cm thick by 27 cm diameter tempered glass plate mounted on the one end separates the planar one-turn induction coil from the plasma. The induction coil is made of copper (with water-cooling) and connected to an L-type capacitive matching network and an rf power generator. The details of the apparatus are found in Ref. [26].

The plasma chamber is evacuated by a diffusion pump, roughly pumped by a rotary pump, giving a base pressure of 9×10^{-6} Torr. The operating gas pressure is controlled by adjusting the mass flow controller. The SF_6 gas pressure is minutely varied in the range of 0.4–1.0 mTorr, and then extended up to 3 mTorr. And a 13.56 MHz generator with a power output of 100–500 W drives the rf current in a flat one-turn coil through the rf power generator and matching network. An rf-compensated cylindrical electrostatic probe (SLP-2000, Plasmart) with a tungsten tip of 0.1 mm diameter and 10 mm length is used to measure the plasma parameters. The probe tip is

located on the axis of the cylinder at 14 cm below the tempered glass plate.

In these experiments, a quadrupole mass analyzer (QMS 200F2, Pfeiffer Vacuum) was mounted on the main chamber and mass spectrometry was performed. All the peaks in the mass spectra being considered, the effective mass of the positive ions was determined as 59.3 amu [20]. The negative ion species have not been identified because it is difficult to attract them in a mass analyzer. Negative ions such as SF_6^-, SF_5^-, SF_3^-, F^- have been observed in inductively coupled SF_6 discharges with similar operating conditions to those in this study [27]. In this work, the effective mass of the negative ions is assumed to be equal to that of the positive ions.

4. Results and discussion

The first step to interpret the I–V curve is to determine the plasma potential and the electron saturation current. The plasma potential is from a maximum of dI/dV curve, as shown in Fig. 1(a). After locating the floating potential, the positive ion part of the characteristic current is raised to the 4/3 power and plotted against V_{pr}, as shown in Fig. 1(b). A straight line is fitted to the part of the curve. Extrapolating to V_f gives an estimate of $I_i(V_f)$.

Fig. 2 shows the positive ion densities deduced by several methods. They are the current balance method, the floating potential method, the three pressure point method (TPM), and the OML theory. The OML assumes large sheath. Since the electron density is very low in the parameter region of this study, the Debye length (and the sheath thickness) is much larger than the probe radius. Therefore, the OML theory is well applicable in determining the positive ion density. The difference in the positive ion density estimated from the floating potential method and that from the current balance at the floating
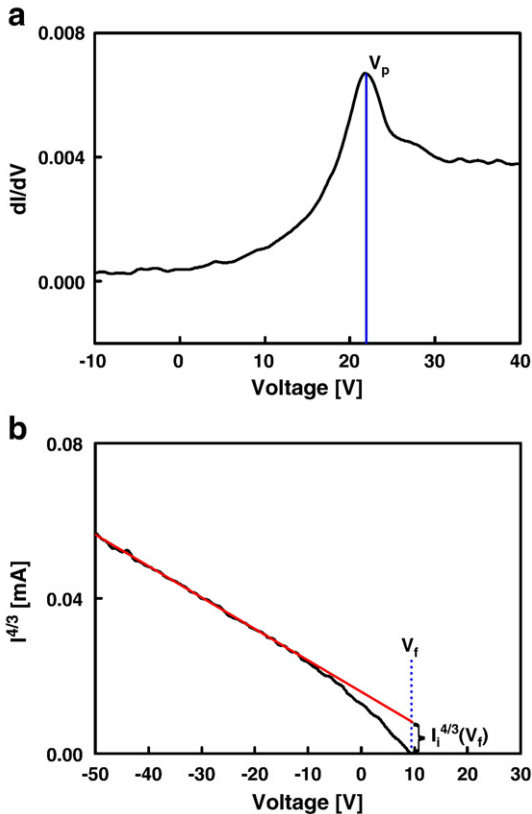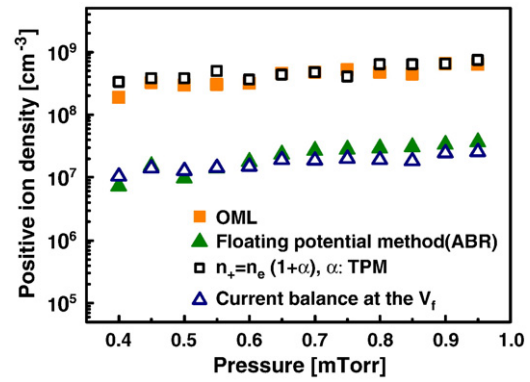

Fig. 2. Comparison of the positive ion density obtained by several different methods (the current balance method, the floating potential method, the three pressure point method, and the OML theory). The pressure range is from 0.4 mTorr to 1.0 mTorr, and $P = 300$ W. (All axes suppress zero point.)

potential is observed to be small. However, these values are relatively lower than those estimated from the OML and the TPM. Since the contribution from the negative ion flux is neglected, the positive ion density might be underestimated in both the floating potential method and the current balance method at V_f. For plasmas having low electron density, the floating potential method cannot provide an accurate estimation of the positive ion density because the sheath length becomes too big. At low densities, I_i^2 fits better to a straight line than does $I_i^{4/3}$, suggesting that some ion orbiting is taking place [21].

Fig. 3 shows the positive ion densities deduced by several methods for the pressure range from 1.0 mTorr to 3.0 mTorr. The value obtained from the current balance method gives the lowest estimation. As before, the positive ion densities estimated from the floating potential method (both based on the Child–Langmuir and the ABR theory) are lower than that obtained by the OML theory. This can be understood in terms of the neglect of the contribution from negative ion flux. Therefore, a reasonable modeling of the negative ion current to the probe is needed for a precise determination of the positive ion density in Eq. (6). However, the difference between the OML results and the results from the floating potential method becomes smaller with an increase in the positive ion density. Although not shown in a figure, it is observed that a comparison of the normalized Bohm flux from Eq.(4) with that from Eq.(6) results in a considerable difference, indicating that the application of $I_{is} = 0.6eAn_+c_s$ at the plasma potential is not appropriate for electronegative plasmas.

Fig. 1. (a) First derivative of the I–V curve. (b) Ion current $I_i^{4/3}$ vs V_{pr}, and a least-squares fitted straight line. The intersection of the line with the vertical line at floating potential V_f yields the value of $I_i(V_f)$. Here $P = 0.7$ mTorr and $P = 300$ W.
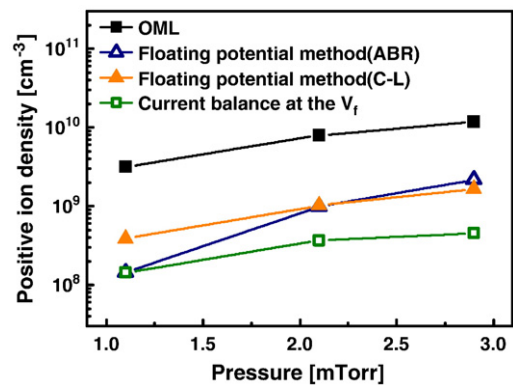

Fig. 3. Comparison of the positive ion density obtained by several different methods (the current balance method, the floating potential methods based on the ABR theory and on the Child–Langmuir theory, and the OML theory). The pressure range is from 1.0 mTorr to 3 ccvc and $P = 300$ W. (All axes suppress zero point.)

5. Conclusion

The electronegative inductively coupled SF_6 discharges have been studied based on electrostatic probe measurements. In principle, the electronegativity can be determined directly from the comparison of experimental positive ion flux with the theoretical Bohm flux. However, it is difficult to deduce the electronegativity from this due to the lack of knowledge on the sheath area A and an inaccuracy associated with the determination of the saturation currents of positive ions and electrons. Instead the positive ion density is obtained by several different methods (the ion saturation current at the floating potential, the positive and negative current balance at the floating potential, and the three pressure point method). All these are compared with the positive ion density obtained by the orbital-motion-limited theory. The positive ion density obtained by the floating potential method and the current balance at the floating potential are relatively lower than those estimated from the OML and the TPM. Since the contribution from negative ion flux is neglected, the positive ion density might be underestimated in both the floating potential method and the current balance method at V_f. An accurate modeling of the negative ion current to the probe is needed for a precise determination of the positive ion density.

6. Acknowledgment

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