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Characterization of an Oxygen Plasma by Using a Langmuir Probe in an Inductively Coupled Plasma

Jong-Sik KIM* and Gon-Ho KIM

Department of Physics, Hanyang University, Ansan 425-795

Tae-Hun CHUNG

Department of Physics, Dong-A University, Pusan 604-714

Geun-Young YEOM

Department of Materials Science and Engineering, Sungkyunkwan University, Suwon 440-746

Kwang-Ho KWON

Department of Electronic Engineering, Hanseo University, Seosan 356-820

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Oxygen plasmas were investigated by using a Langmuir probe for an inductively coupled plasma with various rf powers, 100 ∼ 400 W, and operating pressures, 0.1 ∼ 100 mTorr. The probe current ratio α of the positive and negative currents $(I_+^*/[I_e^* + I_-^*])$ increased with the generation of negative ions and had its maximum value in the pressure region of 40 ∼ 70 mTorr. Also, the operating pressure to achieve the maximum α shifted from the low-pressure region to the high-pressure region with increasing input power because enhanced ion loss through positive-negative ion recombination.

I. INTRODUCTION

Oxygen plasmas have been widely used in many processes for semiconductor manufacturing. In the etching process using CF4, for example, the additive oxygen enhances the liberation of fluorine atoms in the CF4 plasma, resulting in an increased etch rate for the silicon wafer. The oxygen plasma also plays an important role in the ashing and cleaning processes. When oxygen gases are used, negative ions change the plasma potential and the behaviours of the electrons and the ions near the substrate, so it may cause dislocations on the surface during film formation [1,2]. In spite of the need to understand the scaling of the oxygen plasma constituents with the control parameters, investigations have not been progressed sufficiently since the parameter space is much enlarged from that of electropositive plasmas; also, the diagnostics is very difficult in the presence of negative ions.

Unlike electropositive plasma with ions and electrons, an electronegative plasma is composed of three different charges, positive ions, electrons, and negative ions, which were formed from the attachment reaction of electron to the neutral atoms or molecules [3,4]. Kouznetosov *et al.* considered the electronegative discharge plasma to be distributed over three regions in the chamber: the electronegative bulk region, the electropositive edge region, and the sheath region. Hence, the plasma in the electronegative bulk region is composed of negative ions, positive ions, and electrons, so the plasma quasineutrality condition satisfies the charge balance $n_i \approx n_e + n_-$, where n_- is negative ion density, n_i the positive ion density, and n_e the electron density. In comparison, the plasma in the electropositive edge region is composed of positive ions and electrons. Finally, the sheath region has mostly ions [3,5].

Here are the important reactions in oxygen plasmas: The ionization reaction between an oxygen molecule and an energetic electron results in a positive ion $(O_2 + e \rightarrow O_2^+ + 2_e)$. Negative ions are produced by the dissociation reactions of an oxygen molecule and the attachment reactions between an oxygen atom and an electron $(O_2 + e \rightarrow O^- + O)$. The typical loss mechanism in the electronegative oxygen plasma is the recombination reaction between positive and negative ions, $O_2^+ + O^- \rightarrow O_2 + O$. Note that we considered only

*E-mail: jongsik@newton.hanyang.ac.kr

these three reactions in this study, so it was assumed that an increase in the number of negative ions implies a decrease in the number of electrons in the bulk plasma to satisfy the charge balance condition. In recent analytical studies by Lichtenberg *et al.* [6], it was shown that the negative ion production increases with operating pressure and input power. They argued that ion-flux loss to the wall and positive-negative ion recombination are the dominant ion loss mechanisms in electronegative plasmas. They also predicted that the positive-negative ion recombination enhances the ion loss with increasing number of the negative ions, so the number of ions is reduced in plasmas.

As the optogalvanic [7] with spectrometer method [8] for measuring negative ions shows that the Langmuir probe technique by Amemiya [2] is relatively inexpensive and less complicated. Amemiya measured of the negative ion density from the electron energy distribution function which was obtained from the second derivate of the probe data taken below the plasma potential. This method, however, needs great care to be applied in an rf plasma because the plasma potential fluctuation caused by E-mode discharge in the inductively coupled plasma is serious [9,10]. In this study, a common electrical probe was employed to study the generation mechanism of the electronegative oxygen plasma in inductively coupled plasmas (ICP). The probe technique is described in details in the next section.

Here, we present measurements of electronegative oxygen plasmas generated in inductively coupled plasmas and the measurements were carried out over a wide range of operating pressures and rf input powers. The transition of the dominant positive ion loss mechanism is also presented. This paper is organized as follows: Section II describes how to measure the negative ions by using a Langmuir probe in an electronegative oxygen plasma. In Section III, the experimental setup is presented. Section IV presents experimental results and a discussions of the generation of the electronegative oxygen plasma at various powers and operating pressures. Conclusions are presented in Section V.

II. TECHNIQUE OF A LANGMUIR PROBE IN ELECTRONEGATIVE OXYGEN PLASMA

When Langmuir probe data is taken in a plasma with negative ions [2], the I-V trace is composed of three saturated currents: The saturated *positive current* at a deep negative part of the probe bias of a plane Langmuir probe is

$$I_i \approx 0.6 n_i S e \sqrt{\frac{T_e}{m_i}} \ , \tag{1}$$

which is mostly the ion current, and the saturated *negative current* at a positive part of probe bias is the electron current

$$I_e \simeq n_e S e \sqrt{\frac{T_e}{2\pi m_e}} \ , \tag{2}$$

and the negative ion current

$$I_- = n_- S e \sqrt{\frac{T_-}{2\pi m_-}} \ , \tag{3}$$

where e is the electronic charge, S the probe area, and m_i, m_e and m_- the masses of the ion, the electron, and the negative ion, respectively. T_e and T_- are the temperatures of the electrons, and the negative ions, respectively. Note that if the negative ions increase, then the negative ion current increases, and, on the other hand, the electron current decreases, resulting in the negative current (sum of Eq.(2) + Eq.(3)) decreasing due to the negative ion velocity being very small compared to the electron velocity. Furthermore, the current ratio of the positive current to the negative current increases with the production of negative ions.

We introduce new parameter α defined as the current ratio of the positive current to the negative current. Assuming that $T_- = T_+ = T_e/10$ [5], O_2^+ (μ=32) is the major positive ion, and O^- (μ=16) is the major negative ion (see the details in the previous section), so m_i=$2m_-$ [5], and the current ratio α can be expressed as

$$\alpha = \frac{I_+}{I_e + I_-} \approx \frac{1.5 n_+}{240 n_e + 0.2 n_-} \ . \tag{4}$$

As just discussed, α is inversely proportional to the generation of negative ions. Because those assumptions are not always correct in real situations, it is very difficult to obtain an accurate density for the negative ions from this relation.

Considerable care is required to ensure an accurate measurement from the Langmuir probe in inductively coupled plasmas. The plasma potential fluctuation due to the E-mode discharge in the ICP impedes the process of determining the plasma parameters [9,10]. The speed of data acquisition (generally less than 1 Hz) is much slower than the speed of the plasma potential oscillation (13.56 MHz in this study), so the current-voltage signals are time-averaged data, as shown in Fig. 1. Probe data taken in a real DC plasma [shown in Fig. 1(a)] show only one knee of I-V, which clearly appears in the log plot in Fig. 1(c), so the plasma potential is clearly obtained. On the other hand, the probe data taken from an ICP, shown in Fig. 1(b), has a stairway shape with two inflection points [shown in Fig. 1(d)] which correspond to fluctuations of the plasma potential. According to Hershkowitz [11], a probe I-V curve like Fig. 1(b) may be due to several causes: (1) the local ionization effect near the probe, (2) electron or ion beams in plasmas, and (3) rf fluctuating plasmas. To clarify the probe data, we compared the potentials from the Langmuir probe with those from the emissive probe, which is well known tool for measuring the plasma potential accurately without

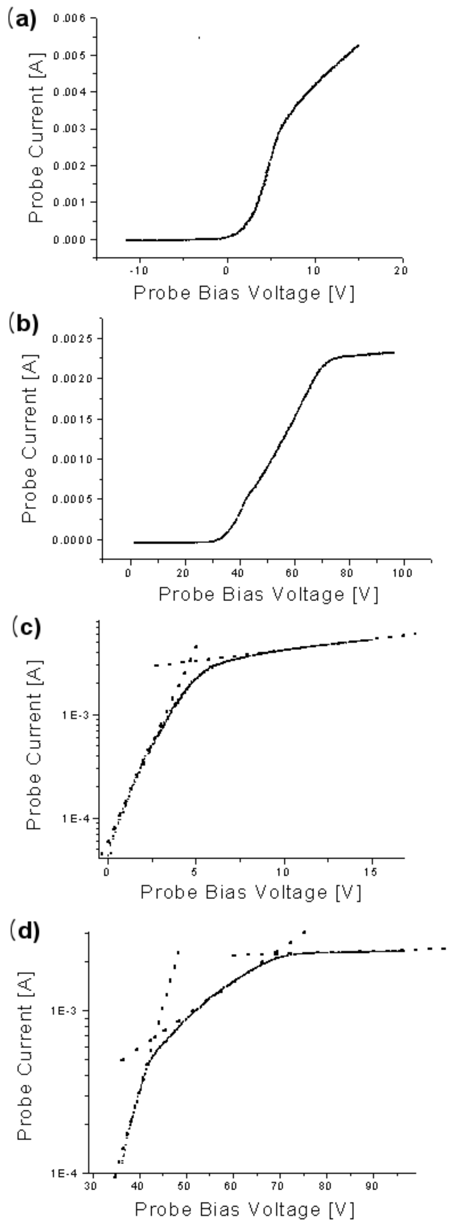

Fig. 1. Langmuir probe data for DC and ICP plasmas: (a) example of Langmuir probe data taken in a DC plasma, (b) example of Langmuir probe data in a ICP plasma, (c) semilog plot of the DC plasma data (arrow indicates the plasma potential), and (d) semilog plot of ICP plasma data (arrows 1 and 2 indicate the low and the high plasma potential, respectively).

being influenced by local ionization or beams. Consequently, the inflection points shown in Fig. 1(b), agreed with the minimum [Vp1 in Fig. 1(d)] and maximum (Vp2 in Fig. 1(d)) plasma potentials, respectively. We concluded that these fluctuation may be caused by the E-mode discharge of the ICP [12]. The plasma potentials Vp1 and Vp2, are chosen from the intersection of the fitted lines in the log plots, as shown in Fig. 1(d). To

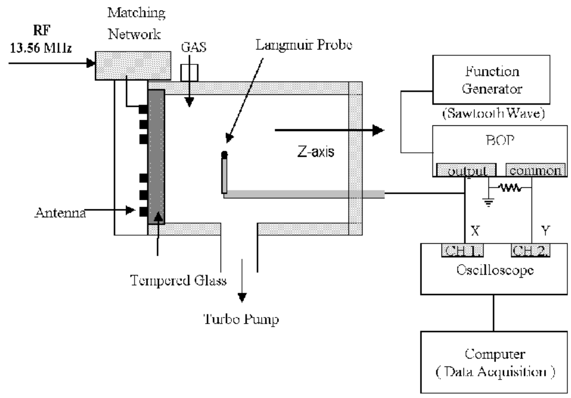

Fig. 2. Schematic diagram of an inductively coupled plasma (ICP) source and Data Acquisition System (DAS) for Langmuir probe diagnostics.

measure the saturated positive current, we fit a straight line to the current data smaller than Vp1 and then took the positive ion current at Vp2. Similarity, a straight line was fitted to the current data larger than Vp2, and the current value at Vp2 was chosen as the total negative current. The electron temperature is obtained from the line fitted to the log data below than Vp1 [as shown in Fig. 1(d)]

III. EXPERIMENTAL SETUP

Characterization of the electronegative oxygen plasma was carried out in the inductively couple plasma source as shown in Fig. 2 Up to 400 W of rf power (13.56-MHz, Henry rf power supply) was delivered to a resonant tank circuit, which consisted of a rectangular-shaped three-turn antenna (300 × 400 mm size and 3.4-uH inductance) and external capacitors. The forward and the reflected powers were monitored by a directional coupler (Bird Wattmeter Model 43), and the reflected power was kept to less than 10oxygen-free copper (square cross-section 10 × 10 mm with 5-mm diameter hole at the center for the path of the cooling water) was located on the top of the tempered glass plate (24-mm thickness). A single-sided planar Langmuir probe (Ta, 6.2-mm diameter) was located 20 cm from the window and at the center of a cylindrical chamber (stainless steel, 500-mm diameter and 600-mm length). The Langmuir probe data were collected by a data acquisition system (DAS in Fig. 2) composed of an oscilloscope with memory function and a personal computer. A bipolar power supply (KEPCO 200) was used to bias the probe. The plasma chamber was evacuated by a turbo-molecular pump (300-ℓ/s pumping speed) and was maintained at a base pressure as 10^{-6} Torr. The ion and the convectron gauges monitored the operating pressure in the chamber, and they were used for low-pressure and high-pressure operations, respectively. The operating pressure was varied widely

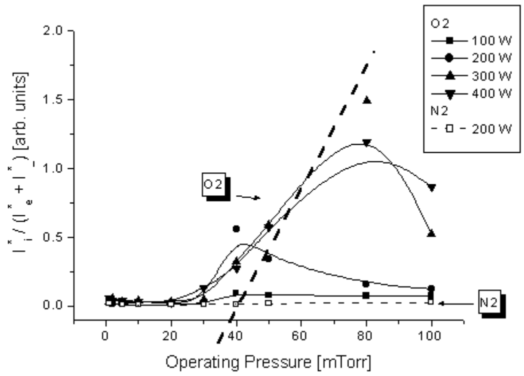

Fig. 3. Current ratio α as functions of the operating pressure and the input power in oxygen (solid line) and nitrogen (dotted line) plasmas. The dashed line shows the variation of the maximum values of the current ratio with input power.

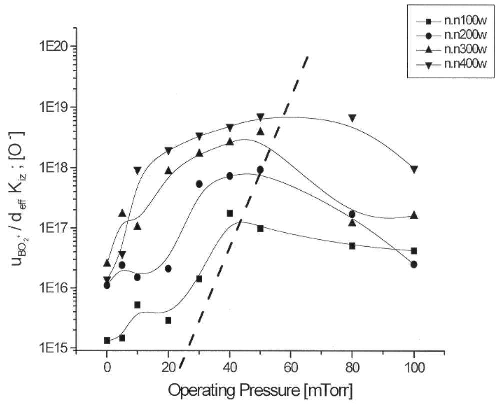

Fig. 5. Distribution of the negative ion density as functions of the operating pressure and the input power in oxygen plasmas. Data were taken from Chung's global model[13] with experimental plasma parameters. The dashed line shows the variation of the maximum values of the current ratio with input power.

from 0.1 to 100 mTorr. Oxygen and nitrogen gases were used to study electronegative and electropositive plasmas, respectively.

IV. EXPERIMENTAL RESULTS AND DISCUSSION

The current ratio α of the positive current to the negative current composed of electron and negative ion currents [as shown in Eq. (4)] was obtained from the Langmuir probe directly. Note that, as mentioned in Sec. II, an increase in α implies an increase in negative-ion generation. Figure 3 shows α as functions of the operating pressure and the input power. The solid line indicates the data taken from the oxygen plasmas, and the dotted line these from the nitrogen plasmas. As shown in Fig. 3, α for the oxygen plasma is much larger and has more variation than the almost constant α, 0.02 over the whole pressure range, for the nitrogen plasma. Note that the nitrogen plasma is well known to be an electropositive plasma. The ratio α of the oxygen plasma varies widely with pressure and increases with input power. The data show that α increases drastically at operating pressures of 40 $\sim$ 70 mTorr, which is approximately 20 times larger than other values. Interestingly, as indicated by the dashed line in Fig. 3, the operating pressure to archive the maximum α shifts to higher values with increasing of input power.

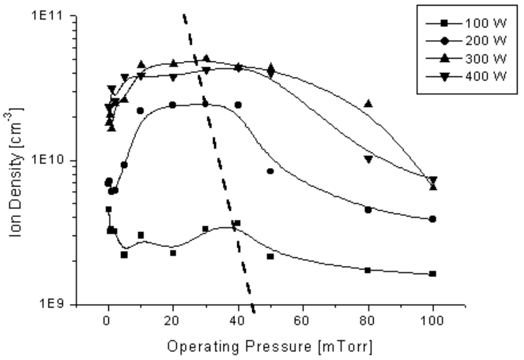

Fig. 4. Distribution of ion density as functions of the operating pressure and the input power in oxygen plasmas. The dashed line shows the variation of the maximum values of the current ratio with input power.

Figure 4 shows the distribution of the ion density as functions of the operating pressure and the input power in the oxygen plasma. The ion density increases with input power and has peak values at operating pressures of 30 $\sim$ 50 mTorr. The operating pressure producing the maximum ion density decreases with the input power, and that is indicated by the dashed line in the figure.

To consider the negative ion generation as functions of the operating pressure and the input power in the oxygen plasma, we used the measured values to apply Chung's global model [13–15]. Following Eq. (32) in Ref. 13, the negative ion density can be scaled as

$$n_{O^-} \approx \frac{R_L u_{BO_2^+} n_e}{R_{rec} d_{eff} n_{O_2^+}} \propto \frac{u_{BO_2^+}}{d_{eff} K_{iz}} \ , \qquad (5)$$

K_{iz} and K_{rec} are the ionization and the recombination rate constants, respectively, $u_{BO_2^+}$ the Bohm velocity of positive ions, R_L rate of recombination and wall flux loss, respectively. The effective plasma length $d_{eff} = \frac{1}{2}\frac{RL}{Rh_L + Lh_R}$, where R is the reactor radius, L is the reactor length, are h_L and h_R are defined as the ratios of the normalized axial and the radial sheath edge densities the densities over the central region of the discharge, respectively. Figure 5 shows the variation of the profile of $u_{BO_2^+}/d_{eff}K_{iz}$ with the operating pressure and input powers, which is proportional to the generation of negative ions. Note that this plot provides just the variation of the negative ions generation with the operating pressure and the input power. Generation of negative ions increases with the power, and maximum generation occurs at operating pressures of 30 ∼80 mTorr. Generation of negative ions increases in the low-pressure region and decreases in the high-pressure region, resulting in the peak value that occurs at a certain pressure and increases with the input power [indicated by the dotted line in Fig. 5]. As discussed in Fig. 4, the pressure producing the maximum positive ion generation decreases with the input power; however, the pressure of maximum negative ion generation increases with the input power. It seems that this behavior is related to the ion loss mechanism in electronegative plasmas. Generation of negative ions increases with the pressure, so the major ion loss mechanism transits from the flux-to-the-wall loss (ion-flux-dominated region) to positive-negative ion recombination loss (recombination-dominated region) with pressure, as predicted in the model [13–15] (see Sec. I). Consequently, with increasing operating pressure, the negative ion generation also increases, and that enhances of the ion loss through the positive-negative ion recombination. Hence, the ion current is reduced and α decreases so that the peak of current ratio α in Fig. 3 takes place at specific pressures of 40 ∼ 70 mTorr to transit the ion loss mechanism.

V. CONCLUSIONS

The characteristics of electronegative oxygen plasma was investigated by using a Langmuir probe. The current ratio α of the ion saturation current to the negative saturation current shows peak values at certain operating pressures, typically 40 ∼ 70 mTorr. In this region, the ion loss is enhanced through the positive-negative ion recombination due to an increase in the number of negative ions. Probe data also show that the generation of negative ions increases with the operating pressure and the input power.

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