Plasma Parameters Obtained with Planar Probe and Optical Emission Spectroscopy

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ABSTRACT

A planar probe and optical emission spectroscopy were employed to analyze parameters in an inductively coupled plasma (ICP). The analyses were performed in Ar, Ar+SF₆ and O₂ plasmas at 40 mTorr. Typical probe results indicate an ion current of 10⁻³ A/cm² and an electron temperature (high energy tail) between 1-2 eV in measurements at high powers. At low powers a distinct discharge regime is observed, typically with low density (low ion current) and high electron temperature. The optical emission studies also showed the presence of these two regimes. Moreover, the electron temperature determination using this diagnostic is also in good agreement with planar probe results.


1. INTRODUCTION

Cylindrical Langmuir probes are widely used to measure plasma electron density and temperature [1]. However, they are known to be mostly sensitive to low and medium energy electrons of the electron energy distribution function (EEDF), with the contribution from energetic electrons being relatively weak and difficult to measure precisely. Also, beside its apparently simple construction (in principle, it can be only a biased wire immersed in the plasma), its employment presents several difficulties. Firstly, in RF-driven technological plasmas, usually there are strong RF fields that interfere with the probe signals. Hence, LC filters should be designed and placed very close to the probe tip [2]. Another problem is the interpretation of the probe data. For some plasma conditions, the simple analysis of the probe characteristic curve (current x voltage) can not be employed [1]. In this situation, the ion orbital motion close to the probe can interfere in the results. A graphical solution method was developed, but it remains a complicated analysis [3].

In order to avoid such difficulties, a new probe method was developed to study technological plasmas [4]. Instead of a thin cylindrical probe, a larger planar probe has been used. With its design, this planar probe avoids the problem of ion orbital motion. Also, it is biased in a different way (with a pulsed RF source) and the data collection is made through a different process (see next section). Also, this probe does not sample the low energy electrons, but the high energy electrons, which can provide complementary data on distinctly different groups of electrons. Moreover, one of the most important features of this probe is that it directly measures the ion flux to the probe (an important information for technological purposes).

Another important plasma diagnostic is the optical emission spectroscopy (OES) from inert gases (such as Ar). In principle, just as for the planar probe, the emission lines from Ar depend mostly on high energy electrons, since the excitation from the ground state level requires a high energy (for Ar, more than 11.5 eV). Therefore, the results of the planar probe and optical emission spectroscopy diagnostics can be compared, analyzing the viability and accuracy of each one.

In this paper, both planar probe and OES diagnostics were employed to study an ICP plasma. A mode transition was clearly identified for all the plasmas when the power was increased. The first mode (at low powers) is mainly a capacitively coupled plasma (since the inductive power is not high enough to promote a significant plasma ionization). Only at high powers, with the reduction of the plasma resistivity, the ionization from the inductive field can be signifi-
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cant, and an inductively coupled plasma is achieved. Even though this mode transition has been known for many years, it is not fully understood, thus it still is object of recent studies [5]. Also, the electron temperature determination using both diagnostics was performed, and their results were compared.

2. EXPERIMENTAL SETUP

The experiments described in this article were performed in an inductively coupled plasma reactor (shown in Figure. 1). The reactor chamber is cylindrical and made of aluminum. The gap between the pyrex window and the electrode was of 9.7 cm, and the chamber diameter is 14 inch. Since the diameter of the planar probe was bigger than the window of the chamber, a small aluminum chamber (14 cm in diameter) was attached to the upper wall of the reactor chamber (at the opposite side of the electrode). With a height of 7 cm, plasma was generated almost entirely inside this small chamber. In the performed experiments, power was only applied to the two turn copper coil - the diameter is approximately 10 cm (the coil is placed in the neighborhood of the window).

The electrostatic planar probe is composed of two main parts: a central disc of 78.54 mm² and a guard ring of 417.83 mm² (to produce a uniform plasma sheath above the central disc). Both parts are biased through different variable capacitors by an RF pulsed generator. Details about the calculations of the plasma parameters using this probe are shown in the next section.

The used optical emission spectrometer (Jobin-Yvon Sofie) has a 35 cm focal length and a 1800 grooves/mm grating, scanning the 200 to 900 nm spectral range. Although almost the entire plasma is formed inside the small chamber, the lower part of the dense plasma region can be viewed below this chamber, allowing the spectroscopy measurements.

The measurements were performed in argon, in oxygen and in Ar + SF₆ plasmas (at concentrations of Ar / SF₆ = 25% / 75%), all at 40 mTorr pressure. For each gas composition the applied power was chosen according to the power in which inductive coupling occurred, so that we could compare capacitively coupled and inductively coupled plasmas, as well as analyze the transition.

3. DIAGNOSTICS METHODOLOGY

The measurement of the ion flux with the planar probe consists of connecting a pulsed RF generator to the planar probe through an external, variable capacitor (placed between the probe and the pulsed RF generator). The pulsed RF voltage applied to the probe is divided in two periods: the ON period (during which an RF voltage is applied, in this study at 12 MHz) and an OFF period (during which the RF voltage is not applied). During the ON period, the probe acquires a negative DC voltage. The probe begins the OFF period with a negative DC bias voltage, which, if sufficiently negative, attracts only positive ions, and therefore, the probe current consists of positive ions only. At the start of the OFF period, the arrival of these ions partially discharges the capacitor and turns the DC voltage less negative, hence the potential barrier for the electrons to reach the probe becomes smaller, and the most energetic electrons become part of the probe current – the net probe current is the ion current minus the electron current. This process ends when the positive ion current through the probe equals the electron current and the probe voltage reaches the floating potential.

The plasma parameters were determined from the behavior of the probe voltage during the OFF period. Figure 2 shows the probe electrical connections. The voltage (\( V \)) over the external variable capacitor \( C_r \) was measured as a function of time (\( t \)). The capacitor \( C_r \) is adjusted to guarantee a uniform and parallel sheath above the central disc, with similar voltage decay in the central disc and in the guard ring. The current (\( i \)) through the capacitor \( C_r \) (connected to the central disc) is equal to the current through the probe, hence:

\[
i = C_p \frac{dV}{dt}.
\] (1)

If we assume that at the beginning of the OFF interval, the electron current through the probe is
negligible, then the current through the probe is composed only of the ion flux. Thus the ion flux can be directly obtained from the initial probe current. Neglecting the effect of negative ions (since in the beginning of the OFF interval their current should be negligible, due to their low energy), and considering the Bohm sheath criterion, the ion flux can be determined from the following equation:

\[ i = e A \Gamma_i = e A n_i c_i, \]  

(2)

where:  
- \( e \) is the electronic unit charge,  
- \( A \) is the probe’s area,  
- \( \Gamma_i \) is the positive ion flux at the interface pre-sheath – sheath,  
- \( n_i \) is the positive ion concentration at the interface pre-sheath – sheath, and  
- \( c_i \) is the ion velocity at the interface pre-sheath – sheath, which is equal to the Bohm speed and is given by [8]:

\[ c_i = \left( \frac{kT_e}{m_i} \right)^{1/2}, \]  

(3)

where:  
- \( kT_e \) is the effective electron tail temperature and \( m_i \) is the positive ion mass (in argon plasmas only the single charged ion was considered, Ar\(^+\), which has a mass of 6.63x10\(^{-26}\) kg; in oxygen plasmas, only the O\(_2^+\) was considered, and its mass is 5.32x10\(^{-26}\) kg, in SF\(_6\) plasmas only the SF\(_5^+\) were considered, and its mass is 2.11x10\(^{-25}\) kg).

From the total current in the probe, one can obtain the electron current through the probe: it is equal to the total current minus the positive ion current. This is facilitated by the fact that because of the planar geometry of the probe, the ion current through the probe is constant during the OFF interval of the voltage applied to the probe. If we assume that the electron energy follows a Maxwell-Boltzmann distribution function, then the electron current through the probe will be proportional to the exponential of the potential barrier between the probe and the plasma [6,7]:

\[ j_e = K \exp\left[ -(V_p - V_s) / kT_e \right], \]  

(4)

where:  
- \( j_e \) is the electron density current through the probe,  
- \( V_p \) is the plasma potential,  
- \( V_s \) is the probe potential, and  
- \( K \) is a constant (dependent on equipment and process).

Then, from the curve of the logarithm of the electron density current versus the potential barrier between probe and plasma, one can obtain the most probable electron energy. If the electron energy distribution function is in fact a Maxwell-Boltzmann distribution, then this curve is a straight line, and its slope is equal to the inverse of the effective electron tail temperature.

These parameters are obtained from the measured probe voltage. However, in reality, the measured voltage contains a lot of noise, which distorts the signal. A previous article describes the method used to transform the noisy signal into a smooth signal [8].

The OES measurements can also determine some plasma parameters, if a proper set of Ar emission lines is selected. Even in the O\(_2\) plasma (as used here), small amounts of Ar (usually a few percent) can be admixed to perform the analysis. In order to analyze the electron temperature, the emissions at 750.4 nm and at 425.9 nm can be used [9]. These emissions have excitation energies (from the ground state) of 13.48 eV and 14.74 eV, respectively. Both have low influence of quenching and radiation trapping and their upper levels (2p\(_1\) and 3p\(_1\), respectively) have small cross section for excitation from metastables [9,10]. So, the comparison of their intensities can be used to calculate the effective temperature of the high energy electrons (here, since their absolute intensities are needed, the spectrometer was calibrated using a calibrated tungsten lamp). The temperature dependence in the ratio Ar 425.9 nm/Ar 750.4 nm is mainly due to difference in their excitation threshold (higher than 1 eV), and is shown in figure 3 [9]. The measurement of these emissions and the use of figure 3 allow the determination of the electron temperature by OES. However, it should be noted that the presence of metastable states can provide an additional channel for Ar atom excitation by low energy electrons. One example is the 2p\(_9\) level (the lowest of the 2p group), which has the largest cross section for excitation from the metastables. So, the 811.5 nm line emission (starting from the 2p\(_9\) state) can be used to analyze the effect of metastables and/or low energy electrons on Ar emission.

Figure 3. Dependence of the intensity ratio Ar 425.9 nm / Ar 750.4 nm with the electron temperature [9].
4. RESULTS AND DISCUSSION

Using the methodology developed in the previous section, the ion current to the probe, the ion density and the electron temperature (high energy tail) were calculated at various powers for each gas composition. In figure 4, the ion current \( \left( e \Gamma_i \right) \) is plotted as function of the applied RF power. For all gas compositions one can see that the ion current suffers an abrupt increase as the power reaches a certain value. This is a consequence of the transition between the two operation modes. The transition occurs at low powers for Ar (63 W), intermediate powers for \( \text{O}_2 \) (240 W) and high powers for \( \text{Ar}+\text{SF}_6 \) (450 W). This matter will be discussed further in the text, at this moment we can point out that the increase of the power required for the transition mode to occur, can be related to two factors: (i) the electronegativity of the gases: electronegative species reduce the plasma electron density and (ii) in molecular gases, electron energy losses are greater due to higher cross sections of their excitation by electrons.

Since the transitions happen at very different powers, for each plasma condition the applied power was normalized by the value at which the transition is observed (see figure 5). Even presenting low values (typically \( 10^{-5} \text{ A/cm}^2 \)) in the capacitive mode, the measured ion current increases with the power. This is a consequence of the increased ionization of the plasma with the absorbed power [6]. Comparing the data before and after the mode transition, the ion current increases about one order of magnitude for Ar and \( \text{O}_2 \) plasmas. For \( \text{Ar}+\text{SF}_6 \) the increase is not so high, but it is still well pronounced (~4 times).

It is important to note that the transitions happen at almost the same ion currents (\( 4-5 \times 10^{-5} \text{ A/cm}^2 \)) for all the plasma conditions. Since the ion current is related to the plasma ion density (remembering that we are neglecting the effect of negative ions), and then, to the plasma electron density, it is an indication that the plasma should reach a minimum electron density so that the transition can occur. This is consistent with our previous description of the mode transitions. As the power is increased, the electron density also increases, reducing the plasma resistivity [6]. When a critical value is reached, the inductive electric field (produced by the oscillating coil current) can promote ionization and the mode transition occurs [11]. Using the results of figure 5, equations (2) and (3), and the calculated electron temperature (as shown later), a similar analysis can be made comparing the calculated ion density in the pre-sheath region (figure 6). However, when \( \text{Ar}+\text{SF}_6 \) is used, the calculated ion density close to the transition is slightly higher than the others. A possible reason for this difference could be a wrong...
estimation of the effective ion mass. When we use the Ar\(^+\) ion mass in eq. (3) for the Ar+SF\(_6\) plasma calculations (instead of the SF\(_5^+\) mass), the result for all the conditions is much closer, being within 30%. However, the full analysis of the accuracy of this estimation and the verification if the ion densities tend to reach the same level before the transition, require more detailed study and is the subject of future work.

In order to explain the results of Figure 4, one can remember that molecular gases (such as O\(_2\) and SF\(_6\)) reduce the electron density due to the high energy losses in excitation of vibrational levels [6]. So, the critical electron density is achieved only at higher powers, increasing the power required for the mode transition. SF\(_6\), a highly electronegative gas (more than O\(_2\)), decreases even more the electron density due to electron attachment. Therefore the transition to inductive coupling occurs at much higher powers.

As shown in Figure 7, the planar probe analysis also indicated the reduction of the electron temperature as the power is increased. Specifically for the O\(_2\) data, the results for low powers (normalized power < 0.5) are not shown, since the data present high errors due to the low measured probe currents. The important point is that quite different behavior was observed in the transition region for the studied gases. Whereas the Ar and O\(_2\) plasmas presented an abrupt reduction of \(kT_e\) in the transition region, the same was not observed for the Ar+SF\(_6\) plasmas.

The increase in the electron density with the transition, detected by the planar probe (Figure 6, except for the Ar+SF\(_6\) plasma where the electron density should not be exactly the ion density), can also be qualitatively observed by OES. It is interesting to note that the Ar 750.4 nm line intensity (Figure 8) does not change notably at the transition, in contrast to the 811.5 nm line. The reason for this is not clear at the moment, and probably it can be attributed to several mechanisms: (i) partial compensation of the effect of electron density increase by the temperature drop at the transition, (ii) stronger heating of the gas in the high-density mode, resulting in lower density of the gas, (iii) OES emission is collected from the lower part of the region where the probe measurements are performed, in this region the rise of electron density may be the smallest. The observed rise of the plasma brightness with the transition can be explained by the increase of other Ar emissions, such as Ar 811.5 nm. Note that the increase of the 811.5 nm line intensity can be related to the increase of both electron and metastable densities.

When the ratio Ar 811.5 nm / Ar 750.4 nm is analyzed (Figure 8), the transition is quite clear, with the abrupt increase of the ratio. Within both modes, this ratio remains almost constant. However, in the inductively coupled mode, the ratio is much higher.
As discussed in [5], the ratio is influenced by the effective electron temperature (decreasing with increasing $kT_e$), but also by the metastables’ concentration in the plasma. So, for pure Ar plasmas, the decrease in $kT_e$ (Figure 7) can explain (at least in part) the observed increase of the Ar 811.5 nm / Ar 750.4 nm ratio with the transition.

Quantitative analyses were performed using the ratio Ar 425.9 nm / Ar 750.4 nm, as discussed above. Figure 9 shows the comparison of the planar probe results (Figure 7) with the OES results. As can be seen, there is a good agreement between both diagnostics. In Ar plasmas the results are slightly higher using OES, but the trend with power is the same for both diagnostics. For oxygen plasmas there is a very good agreement, mainly in the H mode. For Ar + SF₆ plasmas the agreement is also good. The observed differences (such as higher temperatures determined by OES for Ar plasmas, or differences in the E-mode for O₂ plasmas) should be due to deviations from the Maxwellian distribution for the high energy electrons. In other words, the diagnostics do not sample exactly the same electron populations.

During the measurements, the plasma and probe bias were monitored. Their difference is the potential barrier which has to be overcome by electrons to reach the probe. In the beginning of the OFF period (when the electron temperature is calculated) this voltage usually was between 8-10 eV. So, only electrons with energy higher than this value could reach the probe. However, the OES diagnostic samples electrons with higher energy, since there is a threshold excitation for the line emissions (13.48 eV for Ar 750.4 nm and 14.74 eV for Ar 425.9 nm). So, when the results of the two diagnostics are similar this is an indication that the EEDF is close to Maxwellian (at least for the energies probed by the diagnostics). In the same way, in the conditions where the diagnostics presented some differences, this can be attributed to deviations from the Maxwellian distributions of the high energy electrons.

5. CONCLUSIONS

Planar probe and optical emission spectroscopy were employed to analyze the mode transitions in an ICP reactor. The planar probe identified clearly the transition, with a strong increase of the ion flux and ion density. The results showed that a critical density (which should not be exactly the same for all plasma conditions) should be achieved, in order to reduce the plasma resistivity, allowing the transition. When OES results were compared, a qualitative analysis using some Ar line emissions also indicated the electron density increase with the transition. The Ar 811.5 nm / Ar 750.4 nm ratio was a useful indicator of the electron temperature and metastable population changes with the transition. Comparison of the calculated electron temperature using both diagnostics showed a good agreement in most of the situations. The observed difference in some conditions probably indicates the deviation from the Maxwellian distribution of the high energy electrons.
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