Report on Visit to Queen’s University Belfast
by International Training Program

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1. Introduction:

Oxygen discharges have been widely applied in various applications. Early on, oxygen discharges were successfully produced in capacitive rf discharges. More recently, low-pressure high-density discharges have been developed and oxygen plasma has been produced in electron cyclotron discharges, inductively coupled discharges and helicon wave discharges. Oxygen discharges are weakly electronegative and the negative ions (O^-, O_2^- and O_3^-) are expected to contribute significantly to the overall charge balance in oxygen plasma. The presence of negative ions alters the overall discharge phenomena with additional volume recombination loss and a particular spatial distribution of the negative ions which affects the ion flux loss to the wall. O^- is the dominant negative ion; however, a significant fraction (~10%) of O_2^- and O_3^-) ions was present in the plasma. Furthermore, Amemiya *et al* estimate the density of O_2^- ions to be comparable to that of O^- ions and the density of O_3^- ions to be negligible at pressures below 150 mTorr in a parallel plate reactor. The metastable molecule O_2 $(a^1\Delta g)$ is suggested to play significant role in capacitive rf discharges in the pressure range 0.005–0.1 Torr. Detachment by collisions of ions with the metastable O_2 $(a^1\Delta g)$ molecule and Oxygen atoms O (^{3}P) is suggested to be a significant loss process for the negative oxygen ions

It is well known that inductively coupled plasmas operate in two distinct regimes. At lower powers and plasma densities the discharge operates capacitively in so-called ‘E-mode’. When the plasma density increases beyond a critical value the discharge switches to inductive ‘H-mode’. It is accompanied by sudden change in several plasma and circuit parameters such as the electron density and electron energy distribution function (EEDF), the coil current and the optical emission signal. This transition regime between the two modes has a popular parameter space for technological processes, due to better control over the ion energy and flux onto the substrate. The growing interest in E-H transition could be seen by the number of research papers published in recent years.

However, it is important to note that the contribution of various mechanisms to the transition is still complicated or rather ambiguous. Power dependence of optical emission intensity, electron density and temperature, ion density, metastable density, electrical characteristics and magnetic field, has been reported in the literature through the *E–H* transition region

But the detailed knowledge of spatial contribution of various plasma formation mechanisms is crucial, to control essential plasma parameters. Different mechanisms responsible for *E-H* transitions are discussed in a number of theoretical and experimental studies. Rf plasmas in oxygen or in mixtures containing oxygen have wide applications in material processing such as photo resist etching, surface modification, chemical vapour deposition and oxidation. Other interests include thin oxide growth at low temperature by for the fabrication of gate oxide of thin film transistors (TFT). Atomic oxygen has a critical role in plasma volume and surface processes.

It is well known that the admixture of rare gas and oxygen increases the plasma density and processing speed. Several studies have reported the effect of rare gas addition to the Oxygen plasma, by both experiment and modeling. Takechi and Lieberman reported an increase in plasma density and etching rate, with increase in Argon fraction (<50%), in an inductively coupled plasma (ICP) in Ar/O_2, at 20mTorr.

In present study, we measure the negative ions in a Oxygen ICP plasma with laser photo-detachment method. This technique has been well established in un-magnetized and nonsputtering plasmas such as the RF plasma GEC cell.

2. Experimental setup:

The discharge is produced in a GEC reference cell with a five turn planer coil, powered by a 13.56 MHz power supply through a matching box; the electrode diameter is 165.1 mm. The antenna is a five-turn planar coil of 3 mm diameter copper refrigerator tubing (0.75 mm thick walls) that couples to the plasma through a silica window. The top assembly of the source installs in a modified 33.65 mm flange which mates to the Reference-Cell chamber. The source replaces the standard upper electrode assembly. The lower-electrode extension is a disk that rests on top of the standard lower electrode. Bias (dc or rf) can be applied to the lower electrode through the connections that are normally employed to power the standard parallel- plate version of the Cell. A spacer ring in the upper electrode assembly sets the gap between upper and lower electrodes. The antenna-coupling window provides axial optical access to the plasma. Radial diagnostic access is similar to that of the original parallel-plate version of the Cell. The radial dimensions of the source were chosen as a compromise: plasma modelers wished to have electrodes with infinite radial extent (or a nearby solid cylindrical wall), and experimenters wanted large openings for probes and microwave beams. In the present work, the extent of the plasma beyond the 16.5 cm diameter electrode assembly, which was an initial concern, was found to be acceptably small.

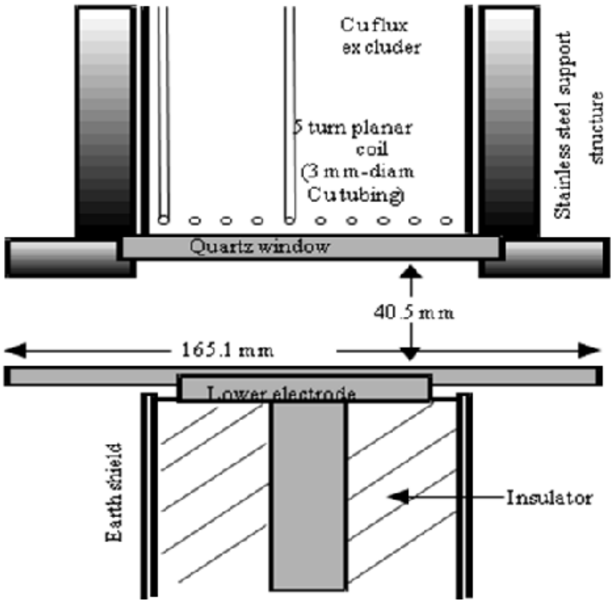

Fig.1. schematic of experimental system.

Technological relevance of electronegative plasmas has already been mentioned. The gas mixtures are often complex and the negative ions play important role in processing or production of the important reactive species, such as O. It is thus helpful to measure the density of O^- , both to enhance understanding the plasma reactions and to connect this understanding to technological processing. No significant flux of O^- leaves the plasma being trapped in the bulk plasma by the positive plasma potential. Thus measurement of O^- densities needs to be performed in the bulk plasma. The detection of negative ions can be divided into two methods, either by conversion of negative ions into electrons by photo-detachment or direct measurement of negative ions using a Langmuir probe. Probe based photo-detachment is the most widely used technique for negative ion density and temperature measurement. It employs a Langmuir probe to detect the increase in electron density due to detachment from negative ions. Nd:YAG laser whose wavelength is 532 nm was employed for this experiment.

3. Results and discussion:

The theoretical photo-detachment fraction is given by:

$$\Delta n_-/n_- = 1-\exp\left(-\frac{\sigma}{h\nu}\frac{E}{S}\right), \quad (1)$$

Here σ means the cross section of the negative ions, ν is the laser frequency, E/S is the laser pulse energy per unit area. According to this equation, the $\Delta n_-/n_-$ ratio saturates at different laser pulse energies depending on the wavelength of the laser. The measurement needs to be calibrated with different parameters, including laser energy, laser diameter, probe bias voltage and so on. Fig.2 shows the comparison of theoretical and experimental results of photo-detachment fraction measurement. One could easily found that either in capacitive mode (50 W, 20 mTorr) or inductive mode (400 W, 100 mTorr), for laser energy less than 25 mJ, the experimental results satisfies equation (1) well; however, for

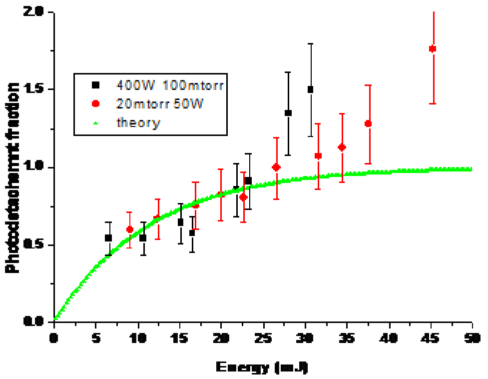

Fig.2. Photo-detachment fraction against energy measured with probe 1.25 cm from lower electrode with laser diameter of 5 mm. The black dot is measured under rf power of 400 W and Oxygen pressure of 100 mTorr; the red dot is measured under rf power of 50 W and Oxygen pressure of 20 mTorr; the green line represents the theoretical value from equation.

laser energy larger than 25 mJ, the lack of saturation of the photo-detached electron current was observed. There are several possible causes for this effect including thermionic electron emission (due to laser and electron current heating - if biased sufficiently above plasma potential) and laser ablation of the probe surface. During ablation the ejected neutrals can become ionized leading to an increasing photo-detachment signal.

Fig.3 shows the effect of probe bias voltage on electronegativity and negative ion current. The system is run in capacitive mode. The fig tells us that when the bias voltage is larger than 45 V, both the electronegativity and negative ion current saturates; therefore we select bias voltage of 45 V for capacitive mode. Similarly, according to Fig.4, we select the bias voltage of 30 V for inductive mode. In addition, laser diameter was also calibrated and selected as 5 mm. Now that we have fixed the probe bias and laser diameter, we could measure the electronegativity. Fig.5 shows the electronegativity measured 2 cm from the ground electrode at rf power of 60 W, 100 W and 160 W respectively. There is a peak electronegaticity at about 13 mTorr. In case of inductive mode (Fig.6), similar situation happens again, which is under the circumstance of inductive mode measured 1.25 cm away from the lower electrode.

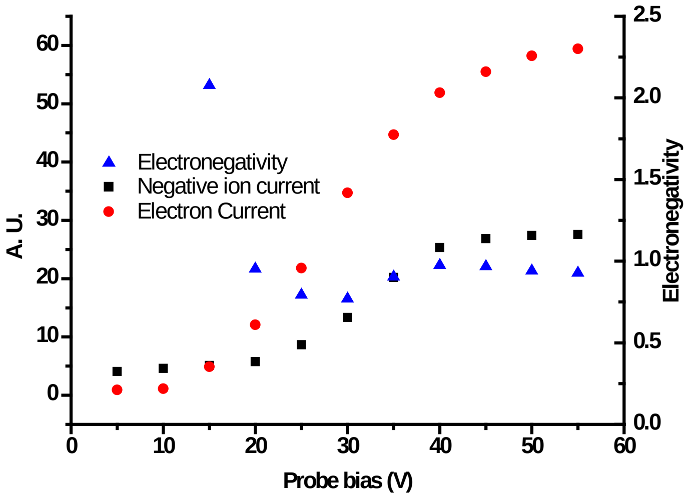

Fig.3. Electronegativity (blue), Negative ion current (black) and electron current (red) against probe bias voltage in capacitive mode.

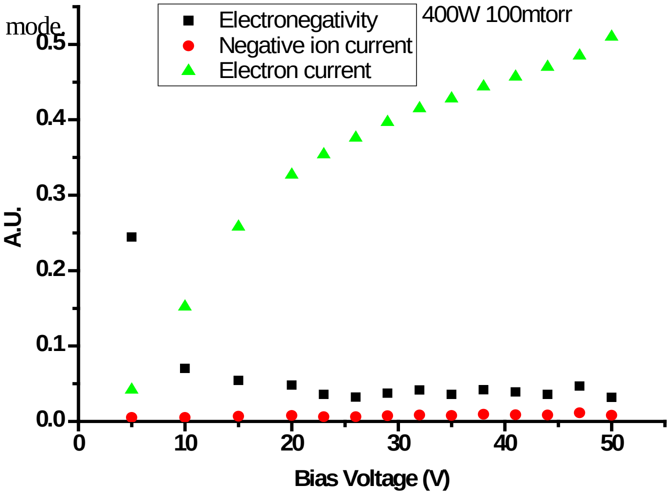

Fig.4. Electronegativity (black), Negative ion current (red) and electron current (green) against probe bias voltage in inductive mode.

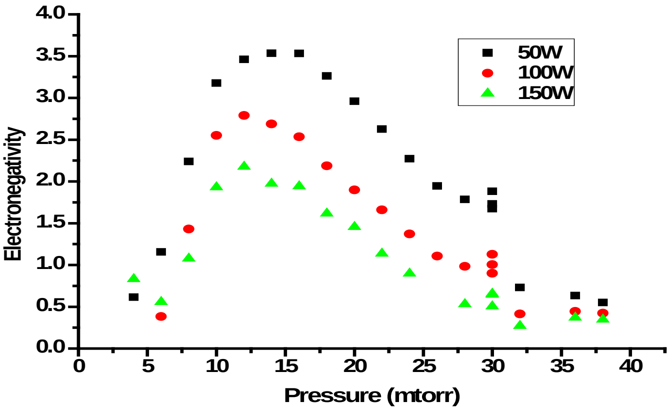

Fig.5. electronagativity against oxygen pressure at different rf power measured in capacitive mode.

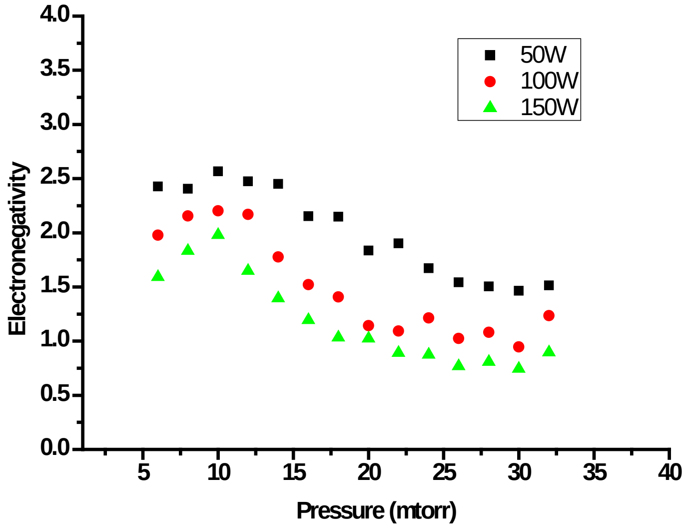

Fig.6. Electronagativity against oxygen pressure at different rf power measured in inductive mode.

The reason for the peak is that the dominant negative ion, O^-, is produced by dissociative attachment of O_2 and destroyed by ion-ion recombination at low pressures. At higher pressures it is lost due to detachment. The destruction between these processes results in a maximum of the negative ion density at a pressure of ~ 12 mTorr in the capacitive mode and 10 mTorr in the inductive mode.

Compared with the simulation results using a global model, as is shown in Fig.7 (black curve), we can see that the measurement results in fig.5 and fig.6 is consistent with the simulation result.

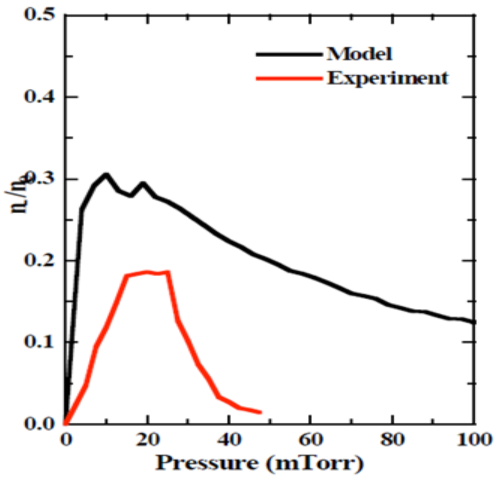

Fig.7 Comparison of experimental and simulation results of the electronagavity against oxygen pressure.

4. Conclusion:

By using a probe based photo-detachment measuring system, we studied the electronegaticity characteristics of Oxygen ICP plasma. Photo-detachment fraction was measured and compared with theoretical value. In order to measure the electronegaticity, we choose proper probe bias and laser diameter under both capacitive mode and inductive mode. The relationship between electronegativity and pressure was plotted and a peak electronegaticity was found at certain pressure. The results are consistent with the simulation results.