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Langmuir Probe Measurements in Electronegative RF Plasma

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Abstract. In this article we present Langmuir probe characteristics measured in hollow cathode plasma jet system powered by RF (radiofrequency) generator on frequency 13.56 MHz. Data were obtained in argon and in argon with admixture of iodine I_2. First results show the effect of negative iodine ions present in plasma.

Introduction

Plasma devices are nowadays widely used for various purposes like thin layer deposition, plasma etching (also called dry etching), plasma chemistry, in lasers etc.

Plasma device can be divided into several categories according to the frequency used for plasma creation. There exist direct current (DC) discharges, radiofrequency (RF) discharges [*Goedheer*, 2000], microwave discharges etc. Plasmas using either pulsed mode or higher frequencies are useful, e.g., for non-conducting thin layers deposition. Such layers can acquire charge in DC discharges and can cause arcing which leads to the layer damage.

In our laboratory experimental devices for thin layer deposition are the subject of research. One of such devices is hollow cathode plasma jet system. It is a device in which the gas is flowing through the nozzle which serves also as a hollow cathode. It is widely used for thin layer deposition [*Tichy*, 2009; *Tous*, 2002].

Besides electropositive plasma containing positive ions, electrons and (usually) neutrals, there exists important group of electronegative plasmas, i.e., plasmas containing also negative ions. Electronegative substances, such as hydrogen H_2, oxygen O_2, fluorine F_2, chlorine Cl_2 and their compounds have substantial electron affinity and can form negative ions either by simple attachment or by dissociative attachment in which the molecule dissociates into negative and neutral particle.

Theory

In this article we describe measurements in argon and in mixture of argon with iodine (electronegative substance) in a discharge created by radiofrequency (RF) generator. Plasma is called electronegative, when the effect of the presence of negative ions is pronounced enough so as to change plasma structure and its behavior.

Structure of electronegative plasma is a bit different than that of electropositive (see Figure 1). In bulk plasma far from walls there is usually real electronegative plasma, then there is electropositive layer and sheath [*Lieberman*, 1994 or *Kouznetsov*, 1996]. This comes from the fact, that ions are much heavier than electrons and have (in laboratory plasmas) substantially lower kinetic energy, so they cannot reach places to which they are attracted because electrons reach such places first. On the other hand when negative particles are repelled from one place, then only particles with high kinetic energy can reach this place — so there will be more electrons than negative ions obviously.

It is more difficult to sustain electronegative plasma than the electropositive plasma due to the fact, that electrons which could serve for ionization are captured by neutral particles in a process called attachment. Therefore more energy must be usually applied to electronegative plasma for plasma breakdown and sustaining.

Processes in electronegative plasmas are: positive ions formation by ionization, negative ion formation by electron attachment, ion loss by positive ion — negative ion collisions, positive ions loss by flow to the wall, electron detachment from negative ions by impact of neutrals, excited particles etc. [*Franklin*, 2001]. Detachment of electrons is more common in, e.g., oxygen plasma than in plasmas containing halogens (stronger bonded electron due to the higher affinity). Positive ions flow to the walls, while negative ions are created and destroyed in volume of plasma.

Experimental device

Measurements were performed in hollow cathode plasma jet system, in which plasma was generated by radiofrequency (RF) generator on frequency 13.56 MHz. Discharge chamber and experimental setup as a whole were described in article of *Pickova* [2011]. Part of the discharge chamber is nozzle through which the gas is flowing and which also serves as a cathode.

Discharge gas is either argon or argon with iodine admixture. Iodine is solid at normal conditions, so iodine evaporator is used, in which iodine crystals are on the porous material through which the part of argon is flowing. Pure argon and argon with iodine are mixed together before they enter the hollow cathode. Gas flow is measured at the outlet of evaporator by heated flow controller. So far we have not yet calibrated the concentration of iodine present in the discharge.

Diagnostics

Our diagnostic method is Langmuir probe — a wire inserted to the discharge. From current-voltage (I–V) characteristics we can obtain plasma potential, floating potential (probe bias at which the net current to the probe is zero) and electron temperature and concentration. There are different ways to obtain these data, so we can have different values of one parameter and must consider, which model of plasma behavior is more realistic.

Data from Langmuir probe measurements are usually quite easy to obtain, but their interpretation is very tricky even for noble gases (i.e., atomic gases which do not form molecules) in a DC non-flowing discharge. More difficulties arise from using radiofrequency driven discharge, more complex gases or magnetic field.

The presence of radiofrequency signal in plasma makes it still more complicated to find basic plasma parameters — plasma potential, concentration and temperature of electrons and electron distribution function (EDF). Radiofrequency is typically between the plasma frequency of ions and of electrons. Therefore lighter electrons usually move with the rapidly changing electric field, while heavier ions cannot follow fast changes of electric field. This leads to the pickup of the radiofrequency signal by the Langmuir probe and to the distortion of I–V characteristics [*Chen*, 2003].

To prevent such distortion of I–V characteristics, we can use RF compensation to suppress the effect of radiofrequency signal being coupled with the probe circuit. RF compensation can be active, passive or hybrid.

Active RF compensation uses feeding the signal of the same frequency as is the driving frequency of plasma, but with different phase and amplitude. The highest possible floating potential is then indication of the successful RF compensation. This method works very well for sinusoidal signals.

Passive RF compensation of the probe is build by LC (i.e., including inductance and capacitance) filter. This filter should be placed as near the probe tip as possible, so usually the elements are small and are inside the probe holder [*Wendt*, 2001]. In addition to RF filter we can use external auxiliary electrode in the vicinity of the probe tip, which is feeding the signal to the probe through the high capacity element [*Annaratone*, 1991].

In our measurements we used Langmuir probe made of tungsten with diameter 100 μm and length 4 mm. Near the probe tip is RF filter. In later experiments external electrode was used.

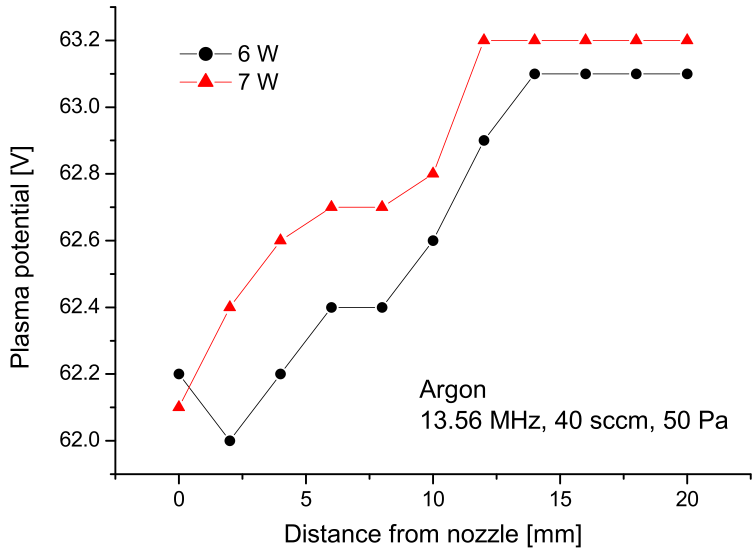

Figure 1. Plasma potential changing with the distance from the nozzle in the argon discharge, near the cathode potential has lower value.

Very serious problem is thin layer deposition on the probe surface. This can lead to the I–V characteristics distortion. This problem often occurs in device dedicated to thin layer deposition and can be diminished by several ways. Normal probe can be bombarded by ions or electrons, when high negative or positive voltage is applied. There is also possibility to heat the probe, so that particles leave the probe surface faster.

Experimental results

Several current–voltage (I–V) characteristics were measured in the argon and in the mixture of argon with iodine. The amount of iodine present in discharge is yet to be calibrated, but its presence is clear from the measured data. Gas flow is measured in sccm, which stands for standard cubic centimeter per minute at standard conditions.

Argon data were measured first to establish measurement techniques before we introduce iodine molecule, which complicates the measurements.

In Figure 1 there are plasma potential data obtained from measurement in argon showing the main parameters changing with distance from the hollow cathode. We can observe expected behavior, that in the vicinity of the cathode the plasma potential is lower than in more distant places.

Below nozzle the effect of negative ions presence is more pronounced. Electron part of the I–V characteristics is bigger for argon than for argon with iodine. Further from nozzle measured electron currents of I–V characteristics tend to be of similar value, although presence of iodine makes it still slightly smaller as we can see in Figure 2.

In Figure 3 we can see changing of electron part of I–V characteristics measured in argon with iodine. As the distance from cathode is increasing current in electron part of characteristics is higher. This is different course to that observed in electropositive argon, where with the distance the electron part of I–V characteristics is getting smaller.

We could also observe effect of thin layer deposition on the probe surface and probe holder (as well as on all inner surfaces of discharge chamber). It lead to the changing of I–V characteristics shape, mainly in the vicinity of floating potential. This problem was solved by high negative bias around −50 V which caused ion bombardment of the probe surface and its cleaning. Ideal use of this treatment was shortly before measurement of each I–V characteristics.

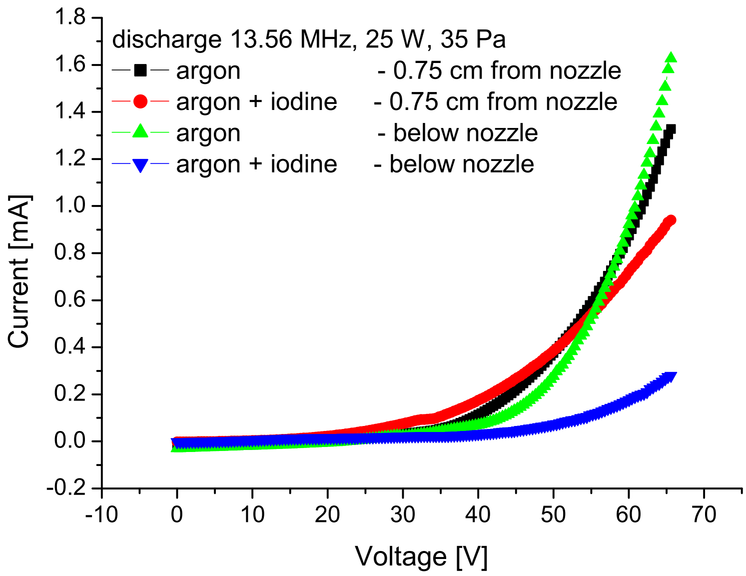

Figure 2. Difference of I–V characteristics measured below nozzle and 0.75 cm from center showing the diminished effect of the negative ions as we move the probe further from the nozzle (i.e., cathode).

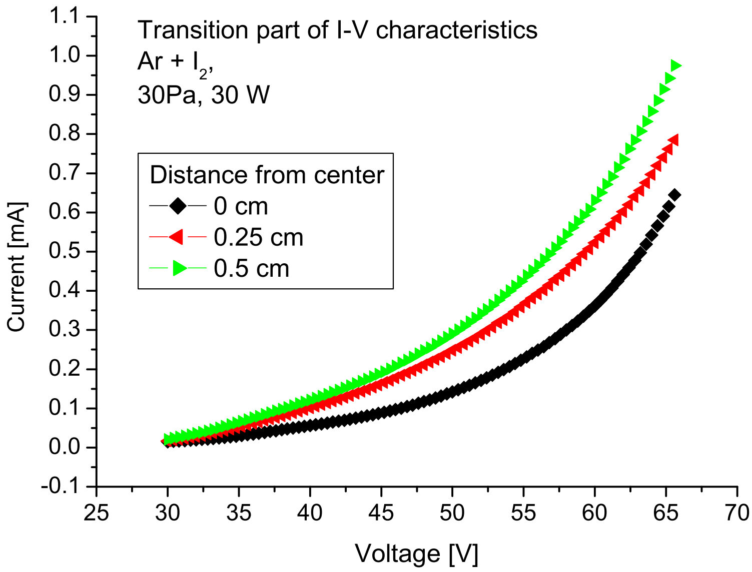

Figure 3. Part of electron current of I–V characteristics measured in mixture of argon with iodine showing that with increasing distance from hollow cathode we observe higher electron current. Data measured in argon only show opposite effect — the further from hollow cathode, the smaller electron current we observe (argon data are not shown here).

Conclusion

I–V characteristics were measured by Langmuir probe in radiofrequency (RF) driven plasma in hollow cathode plasma jet system. First data were obtained in argon so as to see the effect of radiofrequency on the measured I–V characteristics. RF compensation must be used to eliminate RF signal pickup which leads to the I–V characteristics distortion. Very strong effect of thin layer deposition on the probe surface was observed and it was necessary to reduce it by ion bombardment.

Iodine vapors were introduced and the effect of electronegative substance on the shape of characteristics and its changing with the distance from the nozzle was observed. Negative ions cause lower current in electron part of the I–V characteristics. Further from cathode there is higher electron current possibly as a result by detachment of electrons from negative ions caused mostly by collisions with neutrals.

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