## Report on Visit to Texas University at Dallas by International Training Program

## Electrical Engineering and Computer Science, Nagoya University Hironao Shimoeda

I studied on measurements of the electrode voltages of the liquid injection plasma system and evaluations of the plasma potentials at International Center for Advanced Materials Processing (ICAMP), the university of Texas at Dallas, U.S.A. Liquid injection plasmas are expected to be applied to the method of fabricating materials by using solutions such as YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub> which is one of high temperature superconductors. In addition, those are also expected to be applied to a safe and efficient plasma sterilization treatment technique because bacteria dissolved into solutions can be treated. In this study, the measurements of DC self-bias voltage ( $V_{dc}$ ) and RF (13.56 MHz) voltage ( $V_{rf}$ ) of the liquid injection plasma were carried out. Then, the averaged values of  $V_{\text{p}}$  were evaluated by using the outputs.  $V_{\text{dc}}$  is an important parameter to decide the ion energies in the sheath regions. On the other hand, Vp is an important plasma parameter to decide the electron temperature. Generally, V<sub>p</sub> can be measured by probe diagnostics such as Langmuir probe, smart probe, and floating probe. Probe diagnostics can measure various plasma parameters including V<sub>p</sub> easily by inserting a probe into plasmas. However, it is worried about that such as plasma perturbations caused by directly inserted probes would affect the measurements. On the other hand, we can evaluate  $V_p$  with the theoretical calculations from measured V<sub>dc</sub>. That has been proved by Coburn et al. They evaluated V<sub>p</sub> experimentally with the shapes of electrode voltages, which is the sum of  $V_{dc}$  and  $V_{rf}$  [1]. That is why the averaged values of Vp can be evaluated with the methods noncontact to plasmas.

In the beginning of my study, the matching network of the liquid injection plasma system was improved. When the electrode position is changed, the matching condition can be changed. So, it is necessary to correspond the matching circuit to those in order to generate stable plasmas with reducing the loss of the input power and supplying powers to plasmas effectively. The circuit in the matching network is



generally composed of two variable capacitors, shunt capacitor (C1) and series capacitor (C2), and a coil (L). The circuit is used to equal the load impedance in the side of plasma to the characteristic impedance, the value of which is 50  $\Omega$ , with changing the capacitances of the variable capacitors, which is called "matching". As the result, there are no reflected power and power loss. However, we can know the load impedance only by measuring that actually because the impedance depends on such as the plasma impedance and structures of systems. Therefore, in the most cases, people decide the matching conditions in trial and error by adding variable capacitors with wide rage capacitance. In this time, first, the rage of capacitance of two variable capacitors and inductance of the coil used in the matching circuit were measured by using an impedance analyzer. Then, the rage of the load impedance which enables to achieve matching with the variable capacitors was estimated by using a smith chart. With this estimation, fixed capacitors were connected parallel to C1, which changed the available rage of capacitance. As a result, the capacitance can reach to the value of the matching condition with enough extra capacitances to correspond to other conditions. Additionally, the matching conditions were optimized theoretically and experimentally by checking the actual matching condition during plasma generations. Finally, the matching network which can correspond to changes in the load impedance with changing the electrode position was

constructed.

There is the theoretical relation between  $V_{dc}$  and  $V_p$  as shown below [2].

$$V_{p} = \frac{\left(\frac{A_{2}}{A_{1}}\right)^{a}}{\left(\frac{A_{2}}{A_{1}}\right)^{a} - 1} V_{dc} \quad a \le 2.5 \quad (1)$$

where  $A_1$  and  $A_2$  are the areas of the energized electrode and grounded electrode. Therefore, the averaged values of  $V_p$  can be obtained from the values of  $V_{dc}$  and the area of the energized electrode (stage) and surface area of grounded chamber. However, if the chamber is grounded and  $A_1$  of the apparatus is larger than  $A_2$ , there would be parts which don't satisfy the boundary condition of sheath-plasma (ion current is equal to electron current) or aren't clear depending on the structures of that. Because the apparatus used in this study is a typical case of that, the above area ratio is not necessarily correct. But, the ratio of surface areas of each sheathes can be evaluated by estimating the approximate values of  $V_p$  and comparing those with the values obtained experimentally as shown in the example of Coburn *et al.* 

In the liquid injection plasma system, RF capacitively-coupled plasma is generated with a 13.56 MHz RF power supply. The plasma generation conditions of this study are described below. The Ar gas flow rate and pressure inside the chamber were 20 sccm and 16 Pa, respectively. And the RF input power was changed from 1 W to 20 W. The reference electrode position was defined as 0 cm and measurements were carried out with changing the electrode position to 2.5 and 5.0 cm.

Figure 2 shows the circuit for V<sub>dc</sub> measurements. This



Fig. 2 Circuit for V<sub>dc</sub> measurement

circuit is a simple circuit which can take a DC component (V<sub>dc</sub>) out of an electrode voltage by removing a RF component (V<sub>rf</sub>) with a choke coil. This circuit was connected to the output terminal with a BNC terminal of the matching network in parallel. That is why RF leaks and influences of noises would be able to be reduced as much as possible. Then, before conducting the measurements, a sinusoidal wave added some offsets with 13.56 MHz was input into the output terminal of the matching network by using a function generator and it was ensured that only the offsets could be measured after constructing the circuit. From the results of the measurements, it was found that  $V_{dc}$ increased with increasing the RF input power regardless of the electrode positions. This is why it is considered that the result would be caused by increasing the discharge voltage with increasing the RF input power. In addition, the values of  $V_{dc}$  with the electrode position of 2.5 and 5.0 cm were a little smaller than that with 0 cm. Now, there is the relation between the sheath voltages V1 and V2 applied to electrodes with the electrode areas A1 and A2 and each sheath capacitances,

$$\frac{V_1}{V_2} = \frac{C_{sh2}}{C_{sh1}} = \frac{A_2 s_1}{A_1 s_2}$$
(2)

where  $s_1$  and  $s_2$  represent each sheath thicknesses. For a RF plasma, because ion sheathes cannot follow RF voltages,  $s_1$  and  $s_2$  are an approximately-constant value. Accordingly,  $s_1/s_2\sim1$  and it can be assumed that a=1 in the equation (1). Actually, this can be guessed by the experimental results of Coburn *et al* [1]. As a result, from the equation (1), we find

$$V_{p} = \frac{\frac{A_{2}}{A_{1}}}{\frac{A_{2}}{A_{1}} - 1} V_{dc}$$
(3)

The averaged values of  $V_p$  were calculated by substituting measured  $V_{dc}$  into the equation (3). From the result, the averaged values of  $V_p$  also increased linearly with increasing the RF input power. Additionally, the averaged values of  $V_p$ with the electrode position of 2.5 and 5.0 cm were a little smaller than that with 0 cm. It is considered that the changes in  $V_p$  would be caused by changes in the electron area ratio  $R=A_2/A_1$  [1]



Fig. 3 Dependence of electrode voltages on the electrode temperature.

As shown in Fig. 3, Coburn *et al.* estimated  $V_{dc}$  ( $V_t$  in Fig. 3) and  $V_p$  with the wave profiles of electrode voltages and revealed the relation between  $V_1=V_p$  and  $V_2=V_p-V_{dc}$ 

experimentally by changing the ratio of the electrode areas. From the facts that sheath voltages are continuous to  $V_p$  at the boundaries and  $V_p>0$  for a plasma such as an Ar plasma in which positive ions are dominant charged particles, the averaged values of  $V_p$  can be estimated as shown in Fig. 3.

In this study, to improve the accuracy of methods which enable to measure the averaged value of  $V_p$  in noncontact to

plasmas, we aimed to measure the wave profile of the electrode voltage, particularly  $V_{rf}$ . If we can estimate  $V_p$  by using this method, the electrode area ratio (the surface area

ratio of sheathes) could be evaluated by comparing the averaged value of  $V_p$  calculated with the theoretical equation described above. In general, high voltage probes or voltage dividers are used for measurements of an electrode voltage

in plasma. These devices are based on voltage divider circuits which convert high voltages into readily measurable voltages. Therefore, for  $V_{rf}$  measurements, a simple voltage divider circuit was connected to the output terminal of the matching network in parallel. Then, the wave profiles of electrode voltages were measured with an oscillation scope through a BNC terminal. As shown in Fig. 4, the simplest voltage divider circuit is composed of two resistors R1 and R2 connected in series, one of which has comparatively high

resistance value and the other low resistance value. The relation between the voltage  $V_0$  applying to the whole circuit



Fig. 4 Voltage divider circuit



Fig. 5 Equivalent circuit of resistor for high frequency



Fig. 6 Voltage divider circuit for V<sub>rf</sub> measurements

and the divided voltage V<sub>2</sub> applying to R2 is represented as

$$V_2 = \frac{R2}{R1 + R2} V_0 \qquad (4)$$

However, if RF voltages are applied to general electric elements such as resistors, inductance components and capacitance components would affect the characteristic as shown in Fig. 5. For example, we take the resistance value R=1 M $\Omega$ , inductance component L=0.1  $\mu$ H, and capacitance component C=0.1 pF. For a low frequency f=10 Hz, because  $\omega$ L<<R and 1/ $\omega$ C>>R, the impedance value of the element Z-R. On the other hand, for a high frequency f=10 MHz,

because  $\omega L \ll R$  and  $1/\omega C \ll R$ , the impedance value

 $Z\sim 1/\omega C$ . In fact, in the result of measuring the impedance characteristic of a resistor with the resistance value 5.4 M $\Omega$  by using a network analyzer, it was found that the real part of the impedance decreased with increasing the frequency and imaginary part was negative values with thousands because

of influences of capacitance components in MHz zone. Therefore, the voltage divider circuit as shown in Fig. 6 was constructed with two coils which make a role as resistances for high frequency components. In this circuit, the capacitor connected in series removes DC components from electrode voltages and two coils with different inductances divide  $V_{\rm rf}$ 

into a measurable voltage for an oscillation scope. The

relation between the amplitude of the electrode voltage  $V_{rf}$ and that of the voltage wave profile measured with an oscillation scope can be represented theoretically as

$$V_{rf} \approx 1114 * V_0 \qquad (5)$$

Then, a sinusoidal wave added some offsets with 13.56 MHz was input into the output terminal of the matching network by using a function generator and it was ensured that only the sinusoidal wave could be measured after constructing the circuit. However, the relation between  $V_{\rm rf}$  and  $V_{\rm O}$  estimated from the amplitudes of the input voltage and the output voltage was found as

$$V_{rf} \approx 71 * V_0 \qquad (6)$$

From this result, it is considered that capacitance components would affect the characteristic of these coils for RF voltages. The amplitudes of  $V_{rf}$  were calculated by using the actual relation (6) and the averaged values of  $V_p$  were estimated. As a result,  $V_p$  decreased with increasing the RF input power and indicated negative values with input powers of more than 10 W. These results are considered inappropriate from the facts that for plasma such as Ar plasma in which positive ions are dominant charged particles,  $V_p$  must be a positive value and in the above result the electron temperature increased with increasing the input power. This is attributed to the fact that inductive voltages would be induced in the circuit by propagating RF waves across the matching network or RF noises would affect measured wave profiles.

In this study, DC self-bias voltages ( $V_{dc}$ ) of the liquid injection plasma system were measured with changing the electrode position and the knowledge about the ion energy was obtained. In addition, the averaged values of plasma potentials ( $V_p$ ) could be estimated with theoretical equations. However, the electrode area ratio (the surface area ratio of sheathes) couldn't be evaluated because the averaged value of  $V_p$  obtained from the wave profile of the electrode voltage was not compared with that calculated with the theoretical equation described above. To measure more accurate wave profiles of  $V_{rfs}$  it would necessary that capacitors which would have less power losses and sensitive to influences of RF components than coils should be applied to a voltage divider circuit. Additionally, it is also necessary that the frequency characteristics of electric elements are identified with a network analyzer. It is considered that the results have to be compared with those obtained from other methods such as probe diagnostics in order to improve the accuracy of the method noncontact to plasma of  $V_p$  measurements such as described in this study.

Through attending this program, I acquired new knowledge and information by studying on the different research theme from that in Japan. For V<sub>dc</sub> measurements, I learned the difficulty to treat RF components as well as basic knowledge about electric circuit. Though these knowledge and experience are important for the research in Japan, it is hard for us to have opportunities to focus on that basic study and conduct experiments through a trial and error process with constructing circuits actually. Accordingly, I feel that I spent a valuable time. Additionally, because I have never estimate basic plasma parameters such as DC self-bias voltage and plasma potential, knowledge and information which were acquired from these measurements can be applied to future works sufficiently. Concretely, for the study on the plasma treatment processes of the carbon nanomaterial which I'm researching in our laboratory, it can be achieved that the relation between the ion energy and effects of processes would be found and effects of ion energy on the growth mechanism would be evaluated. In addition, there aren't problems that carbon films would be deposited on a probe because the method used in this study enables to measure averaged values of V<sub>p</sub> with noncontact to plasma. Therefore, this method is very effective for evaluations of plasma parameters including Vp during film deposition processes. It is also expected that this method would be applied to apparatus configurations which is difficult to be inserted with probes because the measurements can be conducted by adding simple circuits to a matching network.

I would like to apply the experiences obtained through this program to my lifework including future works and become an engineer or researcher active in the world.

- [1] J. W. Coburn et al., J. Appl. Phys. 43 4965 (1972).
- [2] Principles of Plasma Discharges and Materials Processing