The study of influence of working pressure on mode conversion of argon radio-frequency inductively coupled plasma

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Abstract

Electron temperature and plasma density was measured with change of working air pressure by Langmuir probe at a given power. Radio-frequency (rf) inductively coupled plasma (ICP) discharge mode conversion is determined by optical emission spectrum (OES) with the increase of gas pressure at different power. The results indicate that plasma discharge mode can change from E mode to H mode and spectral intensity appears inverse hysteresis phenomenon with the change of air pressure. The hysteresis width decreases with the increase of power and the hysteresis is no longer present when the power is 420 W and 435 W. Moreover, the jump pressure of E-H transition is gradually reduced with the corresponding increase of discharge power. ICP discharge mode appears E-H-E phenomenon with the increase of air pressure.

Keywords: Inductively coupled plasma; Inverse hysteresis; Optical emission spectrum; Langmuir probe; Mode conversion

1.Introduction

From the first discovery of the inductive discharge in 1884, after more than 100 years of development, the related mechanism of the mode transformation and the nature of the plasma have been gradually understood by the people. But so far there is still not a systematic theory to explain the reasons for the change of mode. In recent years, during the research of the process of the ICP discharge mode conversion, people found that the plasma density would appear hysteresis phenomenon [1-3] when adjusting the rf power back and forth. The reason may be the metastable multi-step ionization [4, 5] or the nonlinear behavior of energy dissipation [6]. However, in addition to the normal hysteresis, we can also observe inverse hysteresis phenomenon under very low pressure [7]. ICP discharge mode changes between the E mode and H mode and plasma parameters also change with the change of the experimental conditions. ICP discharge mode conversion and plasma parameters mutation behavior would bring huge impact on the plasma membrane preparation, plasma etching and plasma stripping process. Therefore, it's meaningful to study the effects of different parameters on the plasma discharge way.

Low temperature plasma is an important tool of material processing. The final material structure and performance are subjected to the plasma temperature, plasma density and electron energy distribution function and so on, which are determined by the experimental conditions such as air pressure, power and flow rate. At present, people focus on rf power, impedance matching network [8] and other conditions on the effect of plasma mode transformation, but no one has a systematic research on the relationship between working pressure and plasma mode conversion. The change of working pressure can cause the change of the plasma density and electron temperature, which makes change of gas load impedance in the chamber and thus affect the current and the electric field intensity in the coil. Therefore, we proposed a conjecture which the

working pressure can also cause the ICP discharge mode transformation and it was further proved by experiment in this paper.

2.Experimental setup

Fig .1 shows that the schematic construction of the cylindrical ICP experimental setup. The height and dimension of reacting cavity are 300 mm and 200 mm, respectively. The height and diameter of the reaction chamber are 300 mm and 200 mm, respectively. We adapt SY type radio frequency power source and SP-II radio frequency adapter which has a power range of 0-1 kW and output frequency of 13.56 MHz as our rf power source. It connects with the induction coils through the matching circuit.

The optical emission spectrum of Ar was measured with a WDS8A-type multifunctional grating spectrometer that is composed of two parts as shown in Fig.1. The inspection window in the discharge cavity was connected with grating spectrometer through optical fiber to measure the intensity of plasma emission spectrum. The monochromator has a raster of 2400 strips mm and a blaze wavelength of 350 nm. Its spectrum measuring range is between 200 nm and 800 nm [9].

1-chamber; 2-optical fiber; 3-Optical Spectrum Analyzer; 4-computer; 5-Langmuir probe system
Fig. 1. The schematic construction of experiment.

Electron temperature and plasma density were measured with Langmuir probe diagnostic technique. The probe, ampere meter and adjustable DC voltage source were connected to each other to form a circuit as shown in Fig. 1. Current in the circuit varies with the change of voltage, so both I-V and lnI-V curves can be obtained [10]. Electron temperature can be achieved by calculating the slope of lnI-V curve, and then combined with a saturated ion current density of the plasma is calculated [11].

3.Spectral diagnosis principle

The plasma spectrum is related to particle radiation transition process. Atoms will produce the excited group A^* , when it impacted with electrons, such as the formula (1) below:

Unstable group A^* will jumps into the low energy ground state or excitation state of A^{**} , the release of the excess energy in the form of photons forming a light emission line at the same time,

such as the formula (2) below:

$$A^* \rightarrow A^{**} + h\nu \quad (2)$$

where $h\nu$ is emitted photon energy. Assume the wavelength of the emission spectrum is λ , the relationship between energy and wavelength is represented by formula (3) below:

$$\Delta E = E_2 - E_1 = \frac{hc}{\lambda} = h\nu \quad (3)$$

where E_2 is higher level of energy, E_1 is Lower level of energy, h is Planck constant, c is speed of light. Each emission line corresponds to a specific wavelength because each particle has a precise energy level. The strongest spectral lines from the first excited state to the ground state of the intrinsic emission lines and the intrinsic spectrum is a group-specific in plasma, therefore, we can infer the group consisting of plasma by the intrinsic spectrum and the intensity of the emission spectrum can reflect the relative concentration of the group of emitting this spectral line.

OES [12, 13] is a non-intrusive measurement method which is used to analyze the spectrum of the plasma by spectroscopic instruments. It can be get the excitation state and ionization state of particles in the plasma, as well as the plasma chemical components and other important information. In this paper, OES for qualitative analysis, in which the plasma discharge mode is determined by scanning plasma emission spectrum, the experiment measured spectral image as shown in Fig. 2.

Fig. 2. The argon spectrum of plasma capacitive coupling discharge mode (E mode) for black line, inductive coupling discharge mode (H mode) for red line.

When the discharge power is 326 W, the relative intensity of Ar characteristic spectrum at 419.5 nm is 57 at pressure of 0.32 Pa and the luminous intensity is lower, which indicates that the plasma is in E mode as shown by black line in Fig. 2. The relative intensity of Ar characteristic peak at 419.5 nm is 530 at 0.34 Pa and the luminous intensity is higher, which indicates that the plasma is in H mode as shown by red line in Fig. 2.

4. Results and discussion

4.1 Probe diagnosis

When ICP discharge power is 357 W, the trends of electron temperature and the plasma density with pressure is shown in Fig. 3 where the black line represents the plasma density and red line indicates the electron temperature. From the graph we can see that the electron temperature and the plasma density increases first and then decreases with the increase of pressure. The maximum value is found in the vicinity of 0.38 Pa and 0.4 Pa, respectively.

Fig. 3. The trend of electron temperature and the plasma density with pressure at the power of 357W.

The reason is that free electrons are accelerated in an electromagnetic field and collide with neutral particles stimulate new free electrons to form a plasma. When the power is constant, with the increasement of pressure, the number of particles increased, which leads to the chance's of collisions between electrons and neutral particles increasing and then the degree of ionization increase, therefore the plasma density increase [14, 15]. The load impedance reduces due to the increase of electron density. This further enhances the current in the coil which makes the field intensity increases that finally raises the electron temperature and the electron energy.

However, as the pressure continues to rise, the increasement of neutral particles, leads the mean free path of electrons to reducing. There is not enough time for electrons to be accelerated in the electromagnetic field and the next collision will be carried out. The average energy of electrons decreases because the energy obtained by electron decreases between the two adjacent collision and the energy loss increases due to the aggravation of collision, therefore, the electron temperature drops. A considerable portion of the electron does not have enough energy to make argon ionization, which means the degree of ionization reduces. Neutral particles, which are the main obstacles, are not conducive to the ionization of the gas, so the plasma density decreases. To synthesize both of these two effects on plasma parameters, which makes the plasma density first

increases to the maximum value and then decreases.

4.2 Spectral measurements

In the case of the discharge power unchanged, we chose six groups of different rf power (357 W, 376 W, 386 W, 400 W, 420 W and 435 W) to explore pressure influence on plasma discharge mode transformation by changing the air pressure, and observed the inverse hysteresis phenomenon. In Fig. 4, the black line shows the relative intensity of Ar characteristic spectrum at 419.5nm changing with the increase of air pressure, and red line shows the relative intensity of Ar characteristic spectrum at 419.5nm changing with the decrease of the pressure.

Fig. 4. The change of relative intensity of Ar characteristic peak with pressure at different power.

Fig. 4(a). shows that the change of relative intensity of Ar characteristic spectrum at 419.5 nm with pressure at 357 W. It can be seen from the Fig. 4(a), the pressure starts to increase from 0.27 Pa and the spectral intensity at this time was 36. As the pressure increases, the intensity increases slowly to 55. During the process, the overall intensity is relatively weak, which can be judged the plasma discharges is in E mode at this time. When air pressure is 0.32 Pa, and spectral intensity mutation to 534, then, spectral intensity increase slowly with the increase of air pressure. Spectral intensity is relatively strong in this process, and the discharge mode is H mode at this time. In the process of the pressure from 0.36 Pa gradually decreasing to 0.27 Pa, the plasma discharge has been in H mode from the beginning to 0.34 Pa. Eventually, as the gas pressure continues to decrease to 0.33 Pa, the spectral intensity suddenly decreases to 57, which means the discharge jumps from H mode to E mode.

We can see from Fig. 4(a) that the inverse hysteresis would occur when pressure is adjusted repeatedly. The pressure point under which E mode jumps to H mode would ahead of that when H mode jumps to E mode. This phenomenon is in agreement with the argument reported previously. Min-Hyong Lee and Chin-Wook Chung got that electron energy distribution function (EEDF) is bi-Maxwellian distribution under E mode while Maxwellian distribution under H mode in lower air pressure [7]. There are much more high-energy electrons under bi-Maxwellian distribution than under Maxwellian distribution. As the ionization energy of Ar atom is 15.76 eV, only part of the high-energy electrons could ionize it, so there is a higher ionization efficiency under bi-Maxwellian distribution. Therefore, when the luminous intensity is the same namely the value of plasma density is the equal, the pressure is lower under bi-Maxwellian distribution than under Maxwellian distribution, which means the pressure jumping from E mode to H mode is lower than that from H mode to E mode, namely the inverse hysteresis.

Fig. 4(b) shows when the discharge power is 376 W, in the process of the pressure increases from 0.26 Pa to 0.34 Pa, the E-H transition occurs when the working pressure is 0.3 Pa. In the process of the pressure reduced to 0.25 Pa from 0.34 Pa, the back H-E transition occurs at the pressure of 0.31 Pa so that an inverse hysteresis is established. Fig. 4(c) shows that there is an inverse hysteresis phenomenon at the power of 386 W, the jump pressure of E-H transition is 0.26 Pa and the jump pressure of H-E transition is 0.27 Pa. There is an inverse hysteresis phenomenon at the power of 400 W, but the jump pressure changes and the hysteresis width becomes smaller as

shown in Fig. 4(d).

Fig. 4(e) shows when the discharge power is 420W, in the process of the pressure increases from 0.18 Pa to 0.25 Pa, the E-H transition occurs when the working pressure is 0.22 Pa. In the process of the pressure reduced to 0.18 Pa from 0.25 Pa, the H-E transition occurs at the pressure of 0.21 Pa. There is no hysteresis phenomenon, which means the inverse hysteresis width is zero.

Fig. 4(f) shows the jump pressure of E-H transition is 0.21 Pa and the jump pressure of H-E transition is 0.2 Pa and there is no inverse hysteresis phenomenon at 435 W.

From Fig. 4 , we summarized discharge mode of E-H transition pressure and the change of the inverse hysteresis width under the condition of different power as shown in Table 1.

discharge power (W)	E-H jump pressure (Pa)	inverse hysteresis width (Pa)
357	0.32	0.02
376	0.30	0.02
386	0.26	0.02
400	0.24	0.01
420	0.22	0
435	0.21	0

Table 1. E-H jump pressure and inverse hysteresis width under the conditions of different power.

Table 1 shows that with the power from 357 W to 435 W, E-H jump pressure from 0.32 Pa is gradually reduced to 0.21 Pa and inverse hysteresis width is gradually reduced until there is no hysteresis phenomenon. This is because under the same conditions, the higher the power the higher the density of plasma and the more the number of particles of gas, and the E mode to H mode can converted in relatively low pressure at high power.

Fig. 5. E-H mode transition pressure under different power

Fig. 5. shows that with the discharge power gradually increased from 326 W to 435 W, the E-H jump pressure is gradually reduced from 0.34 Pa to 0.21 Pa.

To further investigate the influence of pressure on plasma discharge mode conversion under

different power, we selected three groups of fixed power, respectively, 326W, 357W, 386W. In these three groups of power, expanding the scope of the pressure, we continue to increase the pressure to explore how changes in discharge mode and the experimental results are shown in Fig 6.

Fig. 6. The change of relative intensity of Ar characteristic peak at with further increase in pressure at different power.

Fig. 6(a). shows that the intensity of Ar characteristic spectrum at 419.5 nm slow growth from 46 to 57 with the pressure increased from 0.27 Pa to 0.32 Pa at 326 W. When the pressure is increased to 0.33 Pa, the peak mutated to 529, the plasma discharge mode transition from E mode to H mode. With further increase in pressure from 0.34 Pa to 0.35 Pa, the peak back to 58 from 530, which means the plasma discharge mode has changed from the H mode to E mode, and since then it has been in the E mode.

Seen in Fig. 6(a), the discharge pressure range of the H mode is 0.33 Pa to 0.34 Pa at 326 W; in Fig. 6(b), the discharge pressure range of the H mode is 0.32 Pa to 0.41 Pa at 357 W; in Fig. 6(c), the discharge pressure range of the H mode is 0.26 Pa to 0.56 Pa at 386 W.

As can be seen from Fig. 6(d), under the same pressure, high discharge power corresponds to high luminous intensity, which is consistent with former conclusion. In addition, it can be observed that the higher discharge power, the larger range of gas maintain H mode discharge pressure, the lower the power, the smaller the H mode discharge pressure range. And the high power H mode discharge pressure range contains the H mode discharge pressure range of the lower power. When the power is less than 326W, we adjusted the air pressure in any case, the H mode discharge can't established under the experimental conditions. It can be seen: in a certain power, plasma discharge mode would be transformed from E mode to H mode with pressure, and it would be shifted from the H mode to E mode with further increase in pressure, which is called E-H-E phenomenon.

As already described: increasing pressure makes the chance of collisions between electrons and neutral particles increase, which lead to the degree of ionization increase. However, at the same time, the decrease of electron mean free path results in the reduction of electron energy and thus not conducive to the ionization of the gas. Therefore, when the pressure is lower, the smaller of plasma density, the larger of the impedance of the discharge gas, the lower of the output power in a certain power and the plasma discharge is in the E mode. With the increasement of pressure, plasma density increased, which leads to the conductivity's of the gas increasing and then the impedance of the discharge gas reduce, so the output power increases. When the impedance is reduced to a certain value match with the impedance of the rf power supply network, the plasma discharge mode jump into H mode. With further increase in pressure, the ionization degree do not rise rather reduced [16-18], so that the plasma density and temperature decrease, which leads to

the increase of the space gas impedance. Thus the output power decreases and the plasma discharge mode changes from the H mode to the E mode again.

With the increase of discharge power, the pressure range of the gas to maintain the H mode discharge becomes larger. Under the experimental conditions, the impedance of the discharge gas decreases first and then increases within the range of 0.2 Pa to 0.6 Pa. The plasma maintains H mode discharge needing certain output power or output current. To achieve a certain output power, the higher the input power, the greater the variation range of gas impedance. Therefore it can sustain the greater the pressure range of H mode discharge. It can also be explained that the greater the input power, the greater the pressure range of the H mode discharge can be maintained in the case of the same plasma density.

5. Conclusions

In this paper, we show that the change of gas pressure can change the pattern of the plasma discharge. Some conclusions can be obtained as follows:

1 Under certain conditions, the plasma discharge mode can be changed from E mode to H mode with the pressure increase.

2 Spectral intensity appears inverse hysteresis phenomenon with the change of air pressure, and the pressure from E mode to H mode is less than the pressure from H mode to E mode.

3 The hysteresis width decreases with the increase of power. When the power is 420 W and 435 W, the hysteresis is no longer present.

4 The jump pressure of E-H transition is gradually reduced from 0.34 Pa to 0.21 Pa with the discharge power gradually increasing from 326 W to 435 W.

5 If the power remains unchanged, plasma discharge mode appears E-H-E phenomenon with the increase of pressure.

6 The higher discharge power, the greater gas to maintain the H mode of discharge pressure range. The lower the power, the smaller the H mode discharge pressure range. When the power is less than 326 W, it failed to reach H mode discharge if we adjusted the air pressure in any case.

In general, this research could help people to deepen their understanding of the relevant mechanisms and might help people to further study the reasons for the mode conversion.

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