Actions of low- and high-energy electrons on the phase transition between E- and H-modes in an inductively coupled plasma in Ar

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Abstract. It is known that an inductively coupled plasma (ICP) sustained by a radiofrequency current coil has a mode transition and hysteresis characteristics of the internal plasma parameter as a function of the external plasma parameter. We focus on the contributions of low- and high-energy electrons to the phase transition between the capacitive E-mode and the inductive H-mode in an ICP. Our analysis is based on the diagnostics for a time-resolved two-dimensional net excitation rate of short-lived excited atoms, mainly produced collisionally by low- and high-energy electrons by using an intensified charge coupled device optical image. Short-lived excited atoms Ar(2p₁) and Ar(2p₉) with different excitation processes have been employed as optical probes in an axisymmetric configuration of the ICP chamber, driven at 13.56 MHz by an external single-turn current coil at 300 mTorr in pure Ar. The E-to-H transition is characterized by two time constants of electrons: establishment of an axisymmetric distribution by electron diffusion and the accumulation of symmetric high-density electrons in order to sustain the inductive discharge under a weak electromagnetic field. On the other hand, the H-to-E transition is strongly influenced by the presence of a long-lived excited atom (i.e. metastables).

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1. Introduction

A low-temperature inductively coupled plasma (ICP) driven by a radiofrequency (rf) source has a wide range of applications to high technology, such as Si etching on a size of nanometer scale and SiO₂ etching for large-scale integrated circuits [1, 2], micro-electro-mechanical systems fabrication [3], optical emission sources of ultra-microanalyzers [4] and neutral beam sources [5, 6], based on the characteristics of high-density and low-energy electrons. The periodic steady-state plasma and the function of the ICP have been widely investigated experimentally and theoretically during the past few decades [7–10]. In an ICP, it is well known that there are two different modes (E and H) to sustain the discharge in both the cylindrical and planar configurations of the external coil arrangement, and that the mode transition is a function of the external plasma parameter [11–16]. The two operating modes exist and exhibit quite different characteristics in their electrical and optical properties as well as in the internal plasma parameter. In particular, the mode transition in an ICP with two different azimuthal configurations of an external single-looped coil showed different characteristics of coil voltage and emission as a function of dissipated power [17]. Thus, the E-mode depends strongly on reactor geometry, e.g. the configuration and turn number of the current coil. The E–H transition has hysteresis in its electrical property [11] and optical emission [12, 13].

In our previous work, we studied the two- and three-dimensional (2D and 3D) profiles of the net excitation rate in each mode from the emission-selected computerized tomography (CT) images in an ICP source, sustained in a cylindrical quartz chamber driven by an external single-turn current coil at 13.56 MHz [18–23]. CT images showed that the axisymmetric profile in the E-mode is quite different from that of the symmetry in the H-mode [21, 22]. Under these circumstances, few comparisons have been conducted on the effects of low- and high-energy electrons on the transition and the hysteresis. A careful selection of the optical lines is needed on the basis of the collision process between the electron and the neutral molecule. Recently, the E-to-H transition in Ar was observed in 2D space by using a time-resolved ICCD camera [24]. The historical development of the study of the mode transition and the hysteresis in a low-temperature ICP is summarized in table 1. The spatiotemporal transition of the sustaining mechanism in an ICP is still a matter of interest in plasma physics, and this knowledge will make it possible to quantitatively control the stability of ICP for various kinds of applications. It is noted that the macroscopic transition between E- and H-modes in an ICP, such as the external temporal–current–voltage characteristics, is the accumulation of the local current components caused by the electrons. In an axisymmetric configuration of the ICP chamber, the influence of the local electron on both modes will be a key to understanding the kinetic mechanism underlying ICP maintenance.
Table 1. Historical development of mode transition and hysteresis in a low-temperature rf-ICP.

| Year | Observation | Feed gas | Exp./Theor. | Ref. |
|------|-------------|----------|-------------|------|
| 1996 | Hysteresis in $J_{\text{ion}} - P_{\text{rf}}$ | Ar | Exp. | [25] |
| 1996 | Hysteresis in $\Phi_{\text{emit}} - I_{\text{coil}}$ | Ar | Exp. | [12] |
| 1999 | $f(\epsilon)$ in E- and H-modes | Ar | Exp. | [26] |
| 1999 | Modeling of the hysteresis | – | Theor. | [14] |
| 2002 | 3D optical CT of both modes | Ar, Ar/CF$_4$ | Exp. | [21, 22] |
| 2002 | E-to-H transition in $V_{\text{pp}} - P_{\text{rf}}$ | Ar | Exp. | [17] |
| 2004 | Hysteresis in $\Phi_{\text{emit}} - P_{\text{rf}}$ | Ar, Kr, Xe | Exp. | [13] |
| 2008 | $f(\epsilon)$, $n_e$ in transition (hysteresis) | Ar | Exp. | [27] |
| 2010 | 2D-t, E-to-H transition | Ar | Exp. | [24] |

In this paper, we experimentally investigate a phase transition and the contribution of low- and high-energy electrons in an Ar-ICP from a series of optical images in terms of the 2D net excitation rate $R_j(r, t)$ as a function of time $t$ and position $r$ from the ignition of the E-mode and from the stable H-mode.

2. Experimental

An ICP is sustained in a cylindrical quartz chamber with a diameter of 10 cm and a length of 20 cm in the $z$-direction, driven at 13.56 MHz by an external single-turn current coil arranged at the center of the cylinder as shown in figure 1 [19–23, 28]. The advantage of the one-turn-coiled-ICP is in the simplicity of the static field profile to investigate the temporal transition process between E- and H-modes. The temporal change between the modes is realized by applying the external ramped current with the rise and fall rates of the amplitude, 200 A s$^{-1}$. The temporal 2D optical emission, caused by the transition from the upper excited-state $j$ to lower $k$, $\Phi_{jk}(x, y, t)$, is observed by using an intensified charge coupled device (ICCD) camera, set up perpendicular to the single-coil plane. That is, the ICCD camera is mounted 50 cm above the chamber through an interference filter with the full-width at half-maximum 10 nm with respect to the probed emission wavelength. The ICCD is triggered by changing the delay time from the zero point of the ramp current, and is opened for $10 \mu$s for the observation in the form of the time-resolved 2D snapshot. $\Phi_{jk}(x, y, t)$ as a function of external rf-current amplitude will be efficient for investigating the mode transition and the hysteresis in the ICP, because the static and induced electric fields in the cylindrical chamber will be estimated by the external current having the simple one-turn configuration.

Figure 1 shows a schematic diagram of the snapshot measurement for the spatiotemporal E-to-H transition in the present ICP. The optical targets for investigating the transition are short-lived excited atoms, Ar($2p_1$) and Ar($2p_9$), with different excitation mechanisms. That is, the excitation to Ar($2p_1$) and Ar($2p_9$) will be caused mostly by electron impact:

$$\begin{align*}
e + \text{Ar} (1s_0) &\xrightarrow{\epsilon \geq 13.48 \text{eV}} \text{Ar}(2p_1) + e \\
&\xrightarrow{t_{\text{rad}} \sim 21 \text{ns}} \text{Ar}(1s_2) + h\nu(750.4 \text{nm}),
\end{align*}$$

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Figure 1. Experimental arrangement for observing 2D optical emission spectroscopy as a function of time after discharge initiation in a single-turn ICP.

The 2D optical emission image $\Phi_{jk}(x, y, t)$ of the short-lived excited atom with a lifetime of $\sim 10^{-8}$ s corresponds directly to the net excitation rate $R_j(x, y, t)$ at positions $x$ and $y$ at time $t$ [9, 29],

$$R_j(r, t) = n_e(r, t) \int f(\epsilon; r, t) \left( \frac{2\epsilon}{m} \right)^{1/2} N_0 Q_0^j(\epsilon) \, d\epsilon \int f(\epsilon; r, t) \, d\epsilon + n_e(r, t) \int f(\epsilon; r, t) \left( \frac{2\epsilon}{m} \right)^{1/2} N^* (r, t) Q^j_{meta}(\epsilon) \, d\epsilon \int f(\epsilon; r, t) \, d\epsilon,$$  

(7)

and

$$e + Ar(^1S_0) \overset{\epsilon \geq 11.5 \text{eV}}{\rightarrow} Ar(1s_5) + e,$$  

(3)

$$e + Ar(1s_5) \overset{\epsilon \geq 13.08 \text{eV}}{\rightarrow} Ar(2p_9) + e,$$  

(5)

$$e + Ar(1s_5) \overset{\epsilon \geq 13.56 \text{eV}}{\rightarrow} Ar(1s_5) + e,$$  

(6)

$$r_{rad} \sim 30 \text{ns}.$$
Figure 2. Collision cross-section from Ar(1s$_5$) to Ar(2p$_9$) by electron impact. ▲: Mityureva and Smirnov et al [32]; ●: Boffard et al [31].

where $f(\epsilon; r, t)$ is the space- and time-dependent energy distribution of electrons, and $m$ and $n_e(r, t)$ are the mass and number density of the electron. $N_0$ and $N^*$ are the number densities of neutral Ar in the ground state and the metastable state, respectively. $Q^*_j(\epsilon)$ and $Q_{\text{meta}}(\epsilon)$ are the excitation cross-sections by electron impact to the $j$th state from the ground and metastable level, respectively. In particular, the space and time behavior of low-energy electrons during the transition is investigated by using the two-step excitation by way of long-lived metastable Ar(1s$_5$) in reaction (4) (see [30]) and the second term of equation (7). It will be interesting to assess the behavior of the cross-section close to the threshold energy $\epsilon_j = 1.53$ eV in reaction (4). The collision cross section $Q_{1s5}^{2p9}(\epsilon)$ of reaction (4) has been measured by Boffard et al [31, 33] and Mityureva and Smirnov [32]. The cross-section of the spin-conserving excitation process is generally much larger than that of the spin-forbidden process, and it varies slowly as a function of electron energy. This is the case in $Q_{1s5}^{2p9}(\epsilon)$, and the peak value in (4) is 400 times larger than that in (5). $Q_{1s5}^{2p9}(\epsilon)$ has a magnitude of $5 \times 10^{-15}$ cm$^2$ approximated by a unit function with a threshold energy $\epsilon_j$ of 1.53 eV, as shown in figure 2. The energy distribution of electrons in an ICP in an H-mode will decrease to the order of $10^{-2}$ of the peak value at energy greater than the threshold of the direct excitation in (5) [27]. Under these circumstances and the approximation, the net excitation rate $R_j(x, y, t)$, generated by (4) by way of the metastable Ar(1s$_5$), will be estimated as a function of the threshold energy $\epsilon_j = 1.53$ eV. Then, the spatiotemporal transport of the low-energy electrons close to the threshold energy, 1.53 eV, will be visualized at $R_j(x, y, t)$, when the emission from the resultant Ar(2p$_9$) in reaction (4) is observed under the plasma condition of a predominant second term over the first in equation (7). As a result, optical emission spectroscopy offers a great advantage in the diagnostics for low-energy electrons in the discharge in ICP in Ar.

On the other hand, the emission intensity from Ar(2p$_9$) depends strongly on the spatial density $N^*(r, t)$ of the non-emissive metastables Ar(1s$_5$). The metastables are locally generated
Table 2. Time constant related to E-to-H (H-to-E) transition in an ICP ignited at 300 mTorr and 13.56 MHz (E is the local field, N the gas number density and \( \Gamma \) the gas flow).

| Physical quantity       | Typical value(s) | As a function of |
|-------------------------|------------------|-----------------|
| One-period              | \( 7.4 \times 10^{-8} \) | 13.56 MHz       |
| Drift of e              | \( 10^{-7} \)     | \( E/N \)       |
| Diffusion of e          | \( 10^{-7} \)     | \( E/N \)       |
| Drift of p-ion          | \( 10^{-5} \)     | \( E/N \)       |
| Diffusion of \( \text{Ar}^* \) | \( 10^{-3} \)   | \( N \)         |
| Gas residence time      | \( 10^{-1} \) – 1 | \( N, \Gamma \) |
| Lifetime of \( \text{Ar}^* \) | 50                | Impurity        |
| Process time            | 60–100           | Application     |

from the ground state \( \text{Ar}^{(1S_0)} \) by electron impact (3) in the early stage of the discharge and are transported by diffusion with a time constant of \( \sim \) ms. Given the great difference of the transport speed between the electrons and the metastables, the spatial development of \( \text{Ar}^*(r, t) \) less than ms corresponds roughly to the temporal growth of electrons with \( \epsilon > 11.5 \text{ eV} \), while at \( t \) greater than ms the growth time of \( \text{Ar}^*(r, t) \) is controlled by the self-diffusion of metastables (see table 2). It is noted here that the 2D image of the emission from \( \text{Ar}(2p_{\phi}) \), \( \Phi_{jk}(x, y, t) \), represents the space and temporal behavior of the low-energy electrons during the transition between the E- and H-modes, because most of the spatiotemporal change will occur after ms as discussed in section 3. The origin \( (t = 0) \) of the ignition of the E-mode plasma is determined as the time of a frame with a perceptible finite intensity as compared with the previous frames. In the present ICP chamber, the gas residence time is estimated at 0.83 s at 300 mTorr and 50 sccm. After an appropriate pause without external power deposition, 4 s, the procedure is repeated 30 times in order to obtain a series of temporal 2D profiles without statistical scatter. The transition region from the E- to H-mode has a scatter of less than 1 ms, i.e. a deviation of the coil current of \( \pm 0.2 \text{ A} \).

3. Results and discussion

The E-to-H transition after the discharge ignition, and the H-to-E transition after the establishment of the stable H-mode in Ar-ICP at 300 m Torr have been optically studied in the form of the temporal series of the 2D net excitation rates \( R_j(x, y, t) \) of Ar. The 2D image is the integration of the emission (i.e. the rate) along the axial \( z \)-direction of the chamber. In particular, we focus the discussion on the influences of low- and high-energy electrons on the transition and the hysteresis characteristics by a careful selection of the short-lived excited states of Ar. Based on the collisional excitation dynamics described in reaction (1) or (4)+(5) and equation (7), the observed 2D optical emissions from \( \text{Ar}(2p_\phi) \) and \( \text{Ar}(2p_\phi) \) are qualitatively related to the spatiotemporal transport of the high- and low-energy electrons during the transition region in Ar-ICP. It should be noted that each observation for E-to-H and H-to-E is performed under the fixed matching network between the rf-source and the ICP reactor at \( t = 0 \), marked respectively in the blue and orange lines in figures 3 and 4. Also, the 2D images in each row in figures 3 and 4 are normalized for intensity.

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Firstly, we will discuss the time- and space-resolved net excitation rates of (i.e. optical emission intensity from) Ar(2p₁), excited by high-energy electrons with $\epsilon > 13.5$ eV. The initial spatial distribution of the emission will be characterized by the prebreakdown mechanism represented by Paschen’s law in Ar [34] under the static potential distribution along the inner wall. That is, at the beginning of the E-mode, as is expected from our previous static 3D CT image [21, 22], the peak value appears in the vicinity of the current terminal connected through the matching network to the rf-power source, and the net rate leaves a trail close to the inner wall of the cylinder. It is a capacitive discharge to maintain a low-density plasma close to the wall. At each position of the inner wall distributed from a higher to a lower potential along the azimuthal direction under the external rf-current coil, the capacitive rf-plasma is mainly sustained azimuthally by the ionization multiplication by reflected rf-electrons in front of the inner wall with local surface potential. The change in the radial and azimuthal intensity reflects the magnitude of the time-averaged ionization multiplication. At the same time, the wall-sheath width in front of the inner wall spreads out radially as a function of azimuthal position with the decrease in wall rf-potential. In fact, we have already captured the circumstances in the optical 2D image at $t = 0$ in figure 2 in our previous paper [24]. With the passage of time, a radial flow of electrons arises by unidirectional diffusion toward the central part at each azimuthal point as shown at 0–15 ms in figure 2(a) in [24] and in figure 3 in this paper.
The electrons, diffused from the ionization region in front of the wall to the central region, will continue to accumulate under the slow recombination rate between the electron and Ar$^+$ in pure Ar [10]. At $t = 0.5$ and 10 ms in figure 3, a lack of collisional excitation is found in the vicinity of both current terminals, and an almost symmetrical profile is formed with a deviated peak at the central part of the glass cylinder at 15 ms. After the axial symmetry with a peak at the center is completed in the 2D spatial profile of Ar(2p), a further formative time is required to accumulate high-density electrons in order to achieve the E-to-H-mode transition (see figure 3). The time constant for the E-to-H transition is estimated at $\sim 16$ ms, which is the sum of two physical times to establish the axisymmetric distribution and to accumulate a sufficient density of electrons to sustain the discharge by the influence of the electromagnetic field. In fact, the change in the local emission from asymmetry to symmetry in the observed 2D image certainly corresponds to the mode transition from E to H in the ICP. The slight deviation of the center of the H-mode to the opposite side of the current terminals is a result of the influence of the local lack of the induced field caused by the rf-coil. It is noted that the coil current drops at the phase of the E-to-H transition, as reported [35].

Next we will discuss the transition from the H-mode to the E-mode in the ICP by decreasing the amplitude of the external rf-current. In figure 3, the H-to-E transition is shown in the orange line of the spatially integrated emission intensity in reaction (2) after the stable
and symmetrical H-mode plasma is sustained at 19.7 A. The complete symmetrical profile implies that symmetrical high-density electrons in the H-mode will strengthen the induced electromagnetic field up to a magnitude comparable to that of the external rf-coil at 300 mTorr. In our previous CT measurement, we showed the density distribution of Ar(2p1) having a bell-shaped profile with a flat peak of $5.7 \times 10^7$ cm$^{-3}$ at 5 mm in a plane right above the coil. Apart from the coil plane, the radial and axial distributions approach the diffusive profile. As a result, the axially integrated 2D profile has an axisymmetric distribution with a single peak at the center (see the H-mode in figure 3). At $t = 0$, the plasma density is estimated at $5 \times 10^{11}$ cm$^{-3}$ from our previous measurement. As expected, the axisymmetric profile with the peak at the center gradually decreases intensity when the external coil amplitude is decreased at a fall rate of 200 A s$^{-1}$. At the final stage of the discharge extinction, the emission profile returns to the appearance of the E-mode at 45 ms, as shown in figure 3.

The series of transitions observed in terms of the net excitation rate of (or the emission from) Ar(2p9) is shown in figure 4. The external plasma parameters are the same as those in figure 3. We find three discrepancies in the 2D structure between Ar(2p1) and Ar(2p9). One is the difference in the time constant for the formation of the axisymmetry in the E-to-H transition. That is, in Ar(2p9) in figure 4, the axisymmetry forms more rapidly than that in Ar(2p1). Thus will be caused by the influence of the stepwise excitation by way of reaction (4) since the diffusion time of the metastable Ar(1s3) is $\sim$ ms. Therefore, the density distribution $N^*(r)$ of Ar(1s3) provides the key to understanding the 2D profile in the transition. The number density $N^*(r)$, measured by laser absorption spectroscopy, shows a radially M-shaped profile with a peak of $5 \times 10^{10}$ cm$^{-3}$ in front of the wall at $z = 5$ mm from the coil plane, and decreases gradually as a function of axial position. It should be noted that the metastable density of Ar(1s3) is five times that of Ar(1s3) [37, 38]. The typical loss processes of Ar(1s3) are classified collisionally into metastable pooling, electron quenching and neutral quenching, and the maximum rate coefficient, $2 \times 10^{-7}$ cm$^3$ s$^{-1}$, is given by electron quenching [39]. This is the reason for the M-shaped distribution of $N^*(r)$ under the high accumulation of electrons. The typical loss processes of Ar(1s3) under the high accumulation of electrons to $\sim 10^{11}$ cm$^{-3}$ at the center [37]. Under the influence of the M-shaped distribution of Ar(1s3), the 2D image of Ar(2p9) in the E-mode and in the E-to-H mode in figure 4 shows a wider distribution than that of Ar(2p1) in figure 3. This is the second point of difference between Ar(2p1) and Ar(2p9). As a result, the change from asymmetry to symmetry in the 2D profile is faster than that in Ar(2p1).

The great difference in the 2D emission images (e.g. the net excitation rate) between Ar(2p1) and Ar(2p9) is also found in the extinction phase in the H-to-E transition. As described in (4) and (5), the net production rate of Ar(2p9) is the sum of both collision processes of the electron. That is, judging from the lifetime of the metastable Ar(1s3), even at the final extinction phase we have to consider the influence of the finite density of Ar(1s3) on the production of Ar(2p9) by the low-energy electrons. In the presence of the axisymmetric density profile of Ar(1s3) and low-energy electrons, it maintains a symmetric emission profile, as compared with that of Ar(2p1) having an asymmetric profile generated by high-energy electrons with $\varepsilon > 13.5$ eV and localized to both terminals close to the quartz wall. Therefore, the intensity of Ar(2p1) in the E-to-H transition, while there is no such profile in Ar(2p9) in figure 3, reaches a minimum at the initial stage in the E-to-H transition. The integrated curve of $R_j$ of Ar(2p1) in figure 3 reaches a minimum at the initial stage in the E-to-H transition, while there is no such profile in Ar(2p9) in figure 4. Here, we have to consider two time constants for the generation and loss of electrons.
corresponding to the low and high energy. The minimum of $R_j$ in Ar(2p$_1$) in figure 3 is caused by the fast radial diffusion loss of electrons with $\epsilon > 13.5$ eV.

4. Conclusion

The hysteresis characteristics in an ICP are historically discussed in terms of the external current or dissipated power as the external macroscopic plasma parameter [12, 25]. In this paper, we investigate the local electrons and the energy required to induce and to develop the spatiotemporal transition in the ICP by using the integrated information of equation (7). For this purpose, we observed the temporal transition of the ICP from the E- to H-mode in terms of the 2D spatial optical profile of the plasma structure in Ar-ICP at 300 mTorr as a function of time. The 2D plasma structures are discussed in terms of the net excitation rate of the short-lived excited atoms, Ar(2p$_1$) and Ar(2p$_9$), with different production processes dependent on the energy range of electrons. The shorter transit time and the higher rate (i.e. intensity) in the E-to-H transition, and the longer time and higher rate in the H-to-E transition in Ar(2p$_9$) in figure 4 as compared with Ar(2p$_1$) in figure 3, are realized under the two-step excitation, as implied in our previous work where the metastable Ar(1s$_5$) has a broad M-shaped profile with the maximum, $5 \times 10^{10}$ cm$^{-3}$, close to the wall and the shallow minimum, $2 \times 10^{10}$ cm$^{-3}$, at the central axis in an almost uniform electron density, $\sim 10^{11}$ cm$^{-3}$ [37]. The results in figures 3 and 4 actually reflect the spatiotemporal change of electrons with low and high energy, respectively. It is a matter of course that the temporal mode transition is based on the spatiotemporal change of the electron energy distribution and the number density between the capacitive and the inductive discharge [27]. The optical 2D images enable us to understand the sustaining mechanism in both the capacitive and inductive discharges and the related phase transition between E- and H-modes in terms of the effect of the low- and high-energy electrons. Modeling work of the dynamic transition between E- and H-modes, complementary to the present experimental observation, is now being carried out in our laboratory.

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