Measurement of electron density with the phase-resolved cut-off probe method


Citation: J. Appl. Phys. 110, 023304 (2011); doi: 10.1063/1.3586561
View online: http://dx.doi.org/10.1063/1.3586561
View Table of Contents: http://jap.aip.org/resource/1/JAPIAU/v110/i2
Published by the American Institute of Physics.

Additional information on J. Appl. Phys.
Journal Homepage: http://jap.aip.org/
Journal Information: http://jap.aip.org/about/about_the_journal
Top downloads: http://jap.aip.org/features/most_downloaded
Information for Authors: http://jap.aip.org/authors
Measurement of electron density with the phase-resolved cut-off probe method

J. H. Kwon,1 S. J. You,2,a) D. W. Kim,1 B. K. Na,1 J. H. Kim,2 and Y. H. Shin2
1Department of Physics, Korea Advanced Institute of Science and Technology, Daejeon, Korea 305-701
2Center for Vacuum Technology, Korea Research Institute of Standards and Science, Daejeon, Korea 305-306

(Received 26 August 2010; accepted 6 April 2011; published online 22 July 2011)

The phase resolved cut-off probe method, a precise measurement method for the electron density, was recently proposed [J. H. Kwon et al., Appl. Phys. Lett. 96, 081502 (2010)]. This paper presents the measurements of electron density using the method under various experimental conditions (different pressures, powers, chamber volumes, and discharge sources). The result shows that the method is not only in good agreement with the previous method using wave transmittance under various experimental conditions but it is also able to find the cut-off point clearly even under difficult conditions such as high pressure (∼1 Torr), high discharge power, and small plasma volume. The details of the experimental setup, the operating mechanism of the probe method, and the data processing procedure (algorithm) are also addressed. Furthermore, the reliability of the measurement method is investigated by using an electromagnetic field simulation with cold plasma model (CST-Drude model, Computer Simulation Technology). © 2011 American Institute of Physics [doi:10.1063/1.3586561]

I. INTRODUCTION

Cold plasma is widely used in many applications of material processing, such as thin film deposition, etching, implantation, and sputtering.1 In many plasma processes, the electron density is known to be one of the important parameters to control the processing result, because the electron density affects the reaction rates not only in the gas phase chemistry but also in surface chemistry which is directly related to the processing results (deposition, etching, sputtering rates, and film property).2,3 Therefore, it is important to measure the electron density with an accurate and reliable method to control the processing results.

There are several methods to measure the electron density, the most widely used technique being the Langmuir probe.4,5 This method has a good spatial resolution and gives multidimensional information [not only spatial information (x, y, z), but also temporal (t) and energy space information (electron energy distribution function)]. However, the Langmuir probe has proven not to be suitable for measurement in the processing plasma because of some problems such as nonconducting film deposition on the conduction probe, rf noise, and complexity of data analysis.6,7

A method available even for complex plasma conditions is the oscillation probe developed by Sugai et al.,8 first described by Looney and Brown.9 The plasma oscillation method directly gives the absolute electron density directly without complex modeling and assumptions. However, it still has problems in measuring processing plasma:7,8,10–13 the filament used in the electron beam source can induce metal contamination in the processing reactor, the lifetime of the filament is short, and the probe measurement is available only at low pressure. Another probe method, the plasma absorption probe method,7 is applicable to any type of processing plasmas, yet this method is limited to electron densities greater than 1010 cm−3 and to discharge pressures less than 0.5 Torr.14 A recent proposed method called the plasma transmission probe,15 which uses surface waves (SW) propagating along the plasma-sheath boundary, gave a good result for the measurement of local electron density but the selection of the launching frequency of SW in transmission spectrum at low pressure is a problem.

A novel method that employs the transmittance of waves and is usable in various conditions is called the wave cut-off method developed by Kim et al.16–18 To get a transmittance spectrum in this method, a low power frequency-swept signal from a network analyzer is fed to a radiating wire antenna, and the transmitted wave is detected by a detecting wire antenna. This method to get the absolute electron density is very simple and can be applicable in many processing conditions. However, there are also some problems with this cut-off method using transmittance:19 the wave transmission spectrum around the cut-off frequency is sometimes too vague to choose the exact cut-off (frequency) point. In some cases there are lots of cut-off-like signals in the transmission spectrum that are believed to originate from the wave cavity resonances,7 and in the high pressure case, ∼1 Torr, it is hard to choose the cut-off frequency in the transmission spectrum because of the low Q-factor.7

To solve these problems, a new method using the phase difference (φ) between two antennas (called the phase resolved cut-off method) was recently proposed by Kwon et al.20 They found that when the swept frequency is equal to the plasma frequency ω = ωpe, the phase difference between two probes changes abruptly (phase shift) and the frequency for the phase shift slowly changes with low frequency around a hundred Hz in the frequency spectrum (the details and an explanation will be given later). Therefore, using this
phenomenon, they reported that the electron density can be determined very precisely with an error of less than 1%.

This paper investigates the electron density measurement with the phase-resolved cut-off method at various experimental discharge conditions of the ICP discharge. The result shows that the method is not only in good agreement with the previous method using wave transmittance, but is also able to find the cut-off point clearly even in difficult conditions such as high pressure (~1 Torr), high discharge power, and small plasma volume. Moreover, via the measurement in the dc filament discharge with the method, it is shown that the method using the phase difference and its slow time modulation is available not only in ICP discharge maintained by rf power, but also in any other discharge sources maintained by any power source with low frequency power ripples, which are inevitable in all existing power sources. The details for the characteristic of the quadrupole antenna will be published elsewhere\textsuperscript{21}. The background pressure in the reactor is in the 2 × 10\textsuperscript{-6} Torr range, and pure Ar gas (99.9999\%) of 200 SCCM flow is maintained during the operation. The gas pressure is changed from 10 mTorr to 1 Torr and is measured by a Baratron gauge (PDR 2000 dual capacitance diaphragm gauge). To perform the experiments in conditions of small discharge chamber volume, a tripod of 12 cm in height is inserted in the ICP chamber. Furthermore, the measurement is also performed in the tungsten filament dc discharge instead of ICP discharge as shown in Fig. 1(a).

The cutoff probe consists of two antennas as shown in Fig. 2(a).\textsuperscript{16–20} One is the wave radiating antenna and the other is the wave detecting antenna. Both antennas are connected to a network analyzer (HP 8753ET) through the input and output ports, respectively, using a coaxial cable transmission line of 50 Ω. The transmission line is protected from

II. EXPERIMENTAL SETUP

The schematic diagram for the experimental setup is shown in Fig. 1(a). The rf power of 13.56 MHz supplied by an rf amplifier (PFG 1000RF HUTTINGER) is delivered to the ICP antenna through a 50 Ω coaxial cable and L-type matching network (from 150 to 500 W). The plasma is produced with an immersed type quadrupole antenna composed of four quadrant circles and the antenna diameter is 25 cm which is shown in Fig. 1(b). The rf power is fed to the center of the antenna via a central line of the antenna and is evenly distributed to four quadrant circles in the cylindrical vacuum reactor 30 cm in height and 30 cm in diameter. The details for the data processing algorithm, the operating mechanism of the probe method, and the experimental setup are addressed, and a simple reliability test for the method based on the microwave computer simulation is performed.

The article is organized as follows. In Sec. II, we briefly introduce the setup for plasma generation, measurement, and microwave simulation. In Sec. III, the basic theory and methods for the phase resolved cut-off probe used in this article are presented and analyzed. In Secs. IV and V, the experimental results of the cut-off probe for various discharge conditions and calculated results for the reliability test of the method be presented and discussed. We conclude the article in Sec. VI.
FREQUENCY MEASUREMENT

III. BASIC THEORY AND METHODS FOR THE CUT-OFF FREQUENCY MEASUREMENT

The wave cut-off is of the oldest phenomenon in the plasma physics and engineering and widely used in long-distance radio-frequency communication on the earth surface and diagnostic in the plasma.\textsuperscript{5} The cut-off phenomenon can be explained with dispersion relation of the ordinary wave below:

$$\omega^2 = \frac{\omega_{pe}^2}{c^2} + k^2$$

where $\omega$ is a frequency of plasma wave, $\omega_{pe}$ is the plasma frequency, $c$ is the speed of light in the vacuum, and $k$ is a wave number.\textsuperscript{5} By the incident wave ($\omega$), the ordinary wave of the frequency $\omega$ is excited on the plasma periphery and can propagate into the plasma medium depending on the condition for the dispersion relation. For example, when a wave frequency is higher than the plasma frequency of the medium $\omega > \omega_{pe}$, the wave number $k$ is real and the wave of the frequency can propagate in the plasma medium with related phase velocity $\omega/k$. However, while decreasing the wave frequency from the frequency which is higher than $\omega_{pe}$ and if the condition is satisfied $\omega \leq \omega_{pe}$, the wave number ($k$) of the frequency is not real anymore, so the wave cannot propagate into the plasma medium. From the point of view of the plasma, it looks that the plasma may cutoff the incident wave of the frequency, thus it is called the “cut-off” phenomenon of the wave in the plasma. The wave frequency where the cut-off phenomenon starts taking place is called “the plasma cut-off frequency” or the “wave cut-off frequency.” It is noticeable that the cut-off frequency is equal to the plasma frequency which is only a function of the electron density ($\omega_{pe} = \sqrt{n_e e^2/\varepsilon_0 m_e}$). By virtue of this physical relation, it is possible to get the electron density directly by measuring the plasma frequency without any complex sheath and plasma assumptions.\textsuperscript{16}

The plasma density measurement with this cut-off method was developed by Kim et al.\textsuperscript{16} In their experiment, they used two identical antennas (radiating and detecting) instead of Kim’s transmittance has been developed by Kwon et al.\textsuperscript{16} Kwon et al. reported the basic principle of the phase resolved method as follows: as explained before, when a wave frequency applied to the plasma (via radiating antenna) is less than the plasma frequency of the medium $\omega < \omega_{pe}$, a wave of the frequency cannot propagate through the plasma medium but only via the (naturally formed) waveguide between plasma and metal chamber boundary, i.e., the sheath, as a surface wave [see path 1 in Fig. 2(a)].\textsuperscript{15,18,24} However, when the wave frequency is greater than the plasma frequency $\omega \geq \omega_{pe}$, a wave of the frequency can propagate directly through the plasma medium [see path 2 in Fig. 2(a)]. Therefore, in the frequency spectrum, not only does the transmittance signal in Ref. 16 change, the phase difference signal ($\phi$) also changes abruptly.
at $\omega = \omega_{pe}$ as shown in Fig. 7(a), because the favorable wave path length laying between two probes suddenly changes at $\omega = \omega_{pe}$. However, further investigation of the frequency spectrum revealed that the surface wave propagating along the boundaries between plasma and probe holder gives a negligible contribution to the formation of the transmission and phase spectrums. To see the surface wave effect on the spectrums, we investigated microwave simulations for the cut-off probe as we did in our previous paper (under different conditions for the surface wave). Figures 3(a) and 3(b) are cut-off probes with different sheath structures: one has a sheath between two tips, i.e., the surface wave can propagate via the sheath, the other does not have a sheath between tips, i.e., the surface wave cannot propagate anymore. Figures 4(a) and 4(b) are the result of the transmission and phase spectrums, respectively. The Fig. 4 shows that the two results are almost the same, so we can conclude that the surface wave is not the main effect to form the transmission and phase spectrums in our probe structure. Instead of a sudden change of the favorable wave path between two tips, a sudden change of wave characteristic from the evanescence wave to traveling wave at the cut-off frequency is believed to be the reason for the sudden change of the phase at the cut-off frequency. The details for modeling and simulation is beyond the scope of this paper, so they will be presented in forthcoming papers.

To enhance the detection ability of this method for the determination of cut-off frequency, Kwon et al. used an important physical phenomenon: the slowly changing phase difference spectrum signal at the cut-off point with a low frequency of about 100 Hz. This change of phase spectrum at the cut-off point is believed to be an effect of the electron density ripples caused by the applied rf power ripples which is unavoidably generated by the ac-dc power converter (60 Hz) and the rf switching process of transistors (around 100 Hz). To verify this belief, the high voltage probe and the oscilloscope are used to measure the voltage fluctuation on the power fed part of ICP antenna and the result is presented in Fig. 5. As shown in Fig. 5 of the FFT spectrum, there are voltage ripples of low frequency less than or around a hundred hertz on the antenna as we expected.

Normally this rf power ripple is known to be about 0.5% of the applied power, and thus the electron density ripple caused by this rf ripple is supposed to be less than 0.5% of the time averaged electron density because of the rf power balance between electron and ion ($P_{rf} = P_{elec} + P_{ion}$). In the normal plasma of which electron density is of $\sim 10^{10}$ cm$^{-3}$, the ripple of electron density caused by rf power ripple would be $\sim 10^{8}$ cm$^{-3}$ which is too small to be measured by conventional measurement methods and is not believed to affect processing result as chemical reaction rate which is...
proportional to electron density ($n_e$). However, even a small density ripple of a few $10^8$ cm$^{-3}$, which is not important and cannot be resolved in measurements, can lead to the remarkable low frequency oscillation of the cut-off point with the amplitude of $\Delta f \sim$ a few MHz in the frequency spectrum. Figure 6(a) shows the splitting phase different spectrum around the cut-off frequency in repeated measurements at different times. As shown in Fig. 6(a), regardless of the measurement time, there are distorted phase difference spectrums around the cut-off frequency ($\sim 1.35$ GHz) but the distorted phase difference spectra around the cut-off frequency changes depending on the measurement time. It is believed that this phase spectrum change in each measurement is originated from the density ripples caused by rf power ripple of the power supply.

The phase resolved method is used when the sweeping of wave frequency can resolve the fluctuation of the electron density with frequencies around 100 Hz, i.e., the sweeping time around the cut-off frequency is compared with the time of the density fluctuation caused by the rf power ripple. Therefore, in practical measurements, the sweeping time of the wave frequency spectrum is very important to detect the density fluctuation effect. In our case, we swept the 6 GHz frequency spectrum with 1600 data points within 5.4 s. The gap between two data points is 3.7 MHz and the sweeping time for ten data points corresponding to 37 MHz around the cut-off point is about 0.03 s. This time value is quite comparable to the fluctuation time of density [0.01, 0.02, and 0.04 s for each FFT spectrums in Fig. 5 (100, 50, and 25 Hz, respectively)], thus the time resolved phase distortions around the cut-off frequency are measured well as shown in Fig. 6(a). However, if the sweeping time of the frequency spectrum between the points (ten data points around the cut-off frequency) is incomparably longer than that of electron density modulation, the fluctuation effect of the phase difference with time around the cut-off point cannot be measured.

![FIG. 6. (Color online) The phase differences around cut-off probe measured in the repeated measurements with different sweeping speed in the same conditions of Fig. 5. (a) A high speed sweeping $\sim 1.1$ G/s: a condition of 6 GHz frequency spectrum sweeping with 1600 data points in 5.4 s. (b) A low speed sweeping $\sim 0.037$ G/s: a condition of 200 MHz frequency spectrum sweeping with 1600 data points in 5.4 s.](image)

![FIG. 7. (Color online) The various spectrums in the same conditions of Fig. 5. (a) A phase difference spectrum between two antennas [cut-off frequency ($\sim 1.35$ GHz)]. (b) A group delay spectrum which is derivative of spectrum (a) with the frequency $\tau \equiv dq/dt$. (c) A transmittance spectrum. (d) A spectrum $I$ of Eq. (2) calculated from Eq. (2) and the data in Fig. 9.](image)
in any repeated measurements as shown in Fig. 6(b). This is because the fluctuation effect of a few hundreds Hz is temporally averaged during wave frequency sweeping.

For the clear measurement of the distortion point of the phase spectrum (i.e., the cutoff point), the group delay signal which is defined as \( T = \frac{\partial \phi}{\partial t} \) is used. By virtue of the property that the derivative signal has a large value in the discontinuous or distorted spectrum signal regime, the group delay spectrum at the cut-off point can be presented as a magnified signal intensity in the experiment as shown in Fig. 7(b). However, the result of Fig. 7(b) shows that there are still problems pin-pointing the cut-off frequency peak in the phase delay spectrum, because there are many peaks in the spectrum other than the cut-off frequency peak. A phenomenon of the electron density ripple mentioned above is useful to screen out these non-cut-off points generated in this process. Because of the low frequency oscillation of the cut-off frequency, the cut-off point is clearly seen in the group delay spectrum as the frequency point whose spectrum signal amplitude changes most dominantly in the repeated measurement. This frequency point can be easily found with a simple mathematical algorithm\(^{20}\)

\[
I = \sum_i \left( T_i(\omega) - T_j(\omega) \right)^4, \tag{2}
\]

where \( T_i(\omega) \) is the \( i \)th group delay spectrum measured and \( I \) is an arbitrary value representing how much the spectrum value changes for a certain time period (a few tens of milliseconds in our case). Figure 8 presents the group delay spectrums measured at arbitrary different measurement times in repeated measurements. As shown in Fig. 8, it is readily recognized that the cut-off frequency point in the group delay spectrum is clearly represented as the frequency point whose spectrum signal amplitude changes most dominantly in the repeated measurement as we expected before. Therefore, via the mathematical process of Eq. (2), we can get a clear
measurement which has no ambiguity for the electron density measurement and a good agreement with the result of the transmittance method of Fig. 7(c), as shown in Fig. 7(d). The power of 4 in Eq. (2) is an arbitrarily chosen even number to get clean results. It is possible to change other lower or higher numbers. The data processing results with other number powers (2 and 6) are presented in Figs. 9(a) and 9(b), respectively. Figure 9 shows that the higher even number is useful to get more pin-point-like cut-off frequency points in the spectrum.

The overall measurement and data processing of the method are summarized as follows. First we get the phase difference spectrum between two antennas with a wide range frequency spectrum as in Fig. 7(a), which corresponds to the transmittance spectrum in Fig. 7(c). Second, to find the cut-off frequency where the phase difference spectrum has distortion, the spectrum is changed from phase difference to the group delay as in Fig. 7(b). Third, to pin-point the cut-off peak among the other peaks in Fig. 7(b) by using the low frequency oscillation, the repeated measurement is performed as in Fig. 6 and then the measurement data is processed using Eq. (2). Finally, the clear pin-point-like cut-off frequency is measured as in Fig. 7(d).

IV. EXPERIMENTAL RESULTS

The electron densities of the ICP at various conditions (powers, pressures) are measured with two different methods for comparison [the phase resolved method of Eq. (2) and the transmittance method of Ref. 16; the results are presented in Fig. 8]. The results show that, for various conditions, the phase resolved cutoff method can measure the electron density clearly and the result is in good agreement with the existing cutoff method. It is noticeable that even when the transmission spectrum gives unclear measurements for cutoff frequency under certain conditions of power and pressure (see the high power and high pressure, 400 W–100 mTorr, condition in Fig. 10(a) and the 300–500 W at 300 mTorr conditions in Fig. 10(b)], the phase resolved cut-off method gives clear measurements for the cut-off frequency.

In Fig. 11, we summarize the remarkable results of the phase-resolved cut-off method illustrating its advantages when compared with the transmittance method.20 The cut-off frequency cannot be measured clearly with the transmittance method for cases of high pressure (1 Torr) (a), high rf power (b), and small chamber size (c), which is believed to originate from the effect of the low Q-factor (a), and the vacuum and sheath cavity modes in the other cases (b) and (c).7,28 This is a big problem of the previous transmittance method, because it can induce a large uncertainty for the determination of the electron density and a difficulty for the automation of selection for plasma frequency. With the phase resolved method, however, cutoff frequency is determined very clearly even in...
the hard condition for the measurement with the transmittance method as shown in Fig. 11.

To test the accuracy of the proposed method for the measurement of the plasma frequency, we performed a simple uncertainty test for the proposed measurement method by conducting 13 repeated experiments under the same experimental conditions shown in Fig. 12 (50 mTorr, 350 W). The results show that the standard and maximum deviations (used as a maximum difference from the mean value) for the plasma frequency measurement are 0.0025 and 0.0075 GHz, respectively, implying that the plasma frequency can be determined accurately with averaged and maximum uncertainties of 0.19 and 0.28%, respectively, under various experimental conditions. It corresponds to the electron density measured by the proposed phase-resolved method under the same conditions with averaged and maximum uncertainties of 0.38 and 0.57%, respectively. The details of the uncertainty and error bars for the measurement method under various experimental conditions is beyond the scope of this paper, and will be presented elsewhere.

The proposed cut-off method using the phase difference and its slow time modulation is available not only in the RF ICP discharge maintained by rf power, but also in other discharges maintained by any power source with low frequency power ripples, which are inevitable for any electrical power source in practice. To verify this, we performed plasma frequency measurements with the phase-resolved method in the dc filament discharge instead of the RF ICP discharge, and the result is presented in Fig. 13. As show in Fig. 13(a), the plasma frequency is measured well with the phased resolved method and the result is also in good agreement with the transmittance method. The low frequency ripple of dc-filament power is also observed by probing the voltage on the powered tungsten filament as shown in Fig. 1. The measurement of low frequency ripples around 60 Hz is presented in Fig. 13(b).

The measurement mechanism of the phase-resolved cut-off probe can be checked again using a CST simulation with an artificial electron density ripple of 0.5% of averaged electron density. The calculated transmision and phase spectrum of wave frequency at different electron densities (from $0.99 \times 10^{11}/\text{cm}^3$ to $1.01 \times 10^{11}/\text{cm}^3$) are presented in Fig. 14 and the important frequency point regimes including cut-off point and cavity resonance points are presented in Fig. 15 by a magnified image. As shown Figs. 14 and 15(a), the phase spectrums around the cut-off frequency are well divided according to the electron density variation as in the experiment (Fig. 6), in spite of small density variations. This simulation result shows that the method using phase change around cut-off frequency and its slow time variation caused by the electron density ripple is a reliable method for the measurement of the electron density in theory. However, as shown in Figs. 14 and 15, the phase splitting takes place not only at the cut-off frequency (a) but also at cavity resonance frequencies [(b), (c), (c-1), (d)]. Fortunately, the degree of the phase splitting of the cavity resonance point is smaller than those of the cut-off points as shown in Fig. 15. This may be the reason why the cut-off peak is easily identified as the...
whose amplitudes also changed with time like a cut-off peak.

8) by using the simple Eq. (2), although there are others peaks

FIG. 15. (Color online) The magnified simulation result for important frequency points regimes where the density ripple effect can be reflected similar to the cut-off and the cavity resonance frequency points: the magnified phase difference result of (a) $x$ in Fig. 14, (b) $\beta$ in Fig. 14, (c) $\gamma$ in Fig. 14, (c-1) $\psi$ in Fig. 14 with refined phase arrangement, (d) $\delta$ in Fig. 14.

peak whose intensity changes most dramatically (Figs. 7 and 8) by using the simple Eq. (2), although there are others peaks whose amplitudes also changed with time like a cut-off peak.

V. CONCLUSION

In conclusion, as a continuing study of Ref. 20, measurements of the electron density with the phase-resolved cut-off method using the phase difference between two antennas were performed at various experimental discharge conditions. The results showed that the proposed method can measure the absolute electron density precisely within less than a 1% error. The method is not only in good agreement with the previous method using wave transmittance, but it is also able to find the cut-off point clearly even under difficult conditions such as high pressure ($\sim$ 1 Torr), high discharge power, and small plasma volume. Moreover, via the measurement in the dc filament discharge with the method, it was shown that the method using the phase difference and its slow time modulation is available not only for ICP discharge maintained by rf power, but also for any other discharge source maintained by any power source with low frequency power ripples, which are inevitable in all existing power sources, i.e., the method can be applied to all glow discharge sources used in industry. Furthermore, a virtual experiment using an E/M field simulation showed that the method using phase change around cut-off frequency and its slow time variation caused by the electron density ripple is a reliable method for the measurement of the electron density. By virtue of its high precision for the measurement of the electron density, this method is expected to pave the way for a new measurement technique called “plasma chamber matching” which is a highly desired technique in the advanced plasma-semiconductor industry for both fabrication and equipment.

ACKNOWLEDGMENT

This work also sponsored in part by the Korea Ministry of Knowledge Economy (10034836, 10031812-2008-11) and Korea Research Institute of Standards and Science (KRISS).


23See http://www.cst.com for information about E/M wave simulator we used.


