Pure Appl. Chem., Vol. 74, No. 3, pp. 459–464, 2002.

Comparative characterization of high-density plasma reactors using emission spectroscopy from VUV to NIR*

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Abstract: Emission spectroscopy is used to investigate the effect of inert gas mixing in nitrogen plasmas generated in inductively coupled plasma (ICP) and electron cyclotron resonance (ECR) plasma sources. Vacuum ultraviolet (VUV) emission of resonance lines is used to determine concentration of atomic nitrogen while electron temperature is obtained from optical emission spectra. It is found that electron temperature can be either raised or reduced effectively by mixing helium or argon in a nitrogen discharge. Electron-electron collisions and superelastic collisions involving metastable species are key factors in electron temperature tuning.

INTRODUCTION

High-density plasma sources, such as electron cyclotron resonance (ECR) plasma sources, inductively coupled plasma (ICP) sources or helicon plasma sources, are being used in different plasma processing techniques. Their shared features include low operating pressure (below several Pa), high electron densities (up to 10^{13} cm^{-3}), and capability of producing various atomic, molecular, and ion species. But these sources also differ in their excitation frequencies (from RF to microwave), magnetic field strength and configuration, and operating pressure range. These differences can have a significant impact on electron density and its spatial distribution, electron energy distribution function and the generation of active species (both ionic and neutral) and their transport properties in the plasma. Thus, the result of sample treatment varies from one type of reactor to another.

Another consideration in plasma processing is the rate process of active species, and these rates are determined by electron impact and hence electron energy. Several techniques have been proposed to "tune" electron temperature in the plasma [1–4], including electron beam injection [1,2], change of RF excitation mode [3], and the application of a biased grid [4]. On a different front, it has been found that electron temperature in a helium discharge can be "cooled" by adding molecular gases [5,6].

The present work covers two areas: the feasibility of mixing inert gas in a nitrogen plasma as a means of "tuning" (to raise as well as to lower) electron temperature, *Te*, and a comparative characterization of the plasma in ICP and ECR plasmas. The use of inert gas mixing for tuning *Te* also eliminates additional hardware (as in grid or electron beam) or modification of the reactor chamber to accommodate them. Emission spectroscopy is used in the measurement because it is nonintrusive and identifies the prevailing species. Besides a multichannel CCD spectrometer, a VUV monochromator is

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[*] Lecture presented at the 15th International Symposium on Plasma Chemistry, Orléans, France, 9–13 July 2001. Other presentations are presented in this issue, pp. 317–492.

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also used to measure emission of resonance lines of atomic nitrogen, which is responsible for nitridation processes. A Langmuir probe is also employed, when needed.

EXPERIMENT

Figure 1 shows a schematic of an inductively coupled plasma used in the experiment. A quartz tube with a diameter of 17.5 cm is placed on top of a large stainless steel chamber (60 cm ID) with numerous diagnostic ports. A Faraday shield is placed between the quartz tube and RF coil. Up to 1000 W 13.56 MHz RF power can be applied to the plasma. With a 500 L/s turbomolecular pump, the base pressure of the vacuum chamber is as low as 3.8×10^{-5} Pa. Most of the VUV radiation comes from the discharge region (in the quartz tube), where the electron density is highest, and is collected by a movable gold-plated mirror located at the downstream side. VUV light from different vertical chord in the discharge region is reflected by the mirror and is directed into the entrance slit of the VUV monochromator. Its spatial resolution is better than 2 cm.

The ECR plasma source used in the experiment is made by Astex. It has a 2.45-GHz microwave power supply capable of producing up to 1 kW of power. It is also equipped with a pair of electromagnets, which generate a mirror magnetic field. There are four ports available for diagnostics.

The VUV instrument is a McPherson one-meter grazing incidence monochromator. A gold-coated 300 groove/mm grating allows a wavelength range of 50 to 180 nm with a spectral resolution of 0.1 nm. The detector system consists of a photomultiplier tube and sodium salicylate-coated window. A two-stage different pumping system is used, and the pressure inside the VUV monochromator chamber is below 10^{-3} Pa during windowless operation. A UV grade fiber probe and a four-channel CCD spectrometer are used to collect visible spectra along the radial direction. This system is intensity calibrated so that line-ratio technique can be employed to obtain *Te* in the plasma. In particular, a pair of lines from nitrogen's first positive series (762.5 nm) and first negative series (391.4 nm) is used to determine *Te* in the plasma. The electron energy distribution is assumed to be a Maxwellian. Satisfactory agreement of the *Te* measurement has been obtained by the line-ratio technique and a Langmuir probe under plasma conditions relevant to this work. Electron density, *Ne*, is obtained by the Langmuir probe.

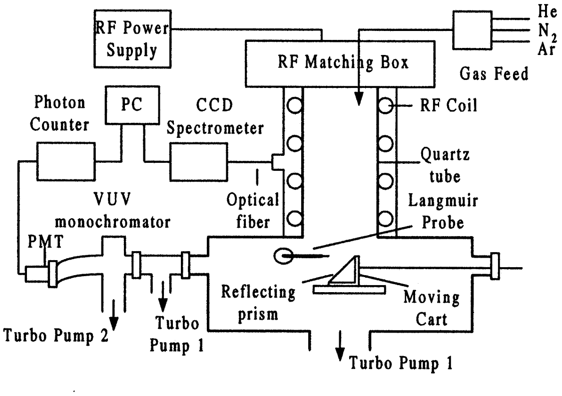

Fig. 1 A schematic of an inductively coupled plasma reactor (MPS-2000).

RESULTS AND DISCUSSION

Figure 2 shows measured change of *Te* (by line-ratio technique) and electron density, *Ne*, in a nitrogen plasma when the concentration of argon and helium gas is varied. As the He partial pressure is increased (the left side of Fig. 2) while keeping the total pressure at 0.7 Pa, *Te* increases, more rapidly at higher partial pressure. Meanwhile electron density decreases. When argon is added to the nitrogen plasma (right side of Fig. 2), *Te* decreases while electron density increases, more rapidly at higher argon partial pressure.

The change of *Te* with partial pressure of the inert gas depends on the size of the "hot tail" in the electron energy distribution function (EEDF). In the helium–nitrogen mixture when electron density is relatively low, the tail is generated by He metastable states and superelastic collisions [7–9]. The tail is enlarged by adding more helium in the discharge and as a result, an "effective *Te*" increases. The tail can also be generated by nitrogen metastable states in a pure nitrogen plasma [10]. However, when argon is added in the discharge, there is a sharp increase in electron density and hence a higher electron–electron collision frequency, which always tend to deplete electrons in the "hot tail" and the EEDF relaxes to Maxwellian. As a consequence, *Te* decreases. The process is similar to that by injecting an electron beam [1] and can be described by the Global Model [11].

Figure 3 shows the corresponding (to Fig. 2) relative change of N I line intensity (120.0 nm, ^4P–^4S) and the line-integrated density of atomic nitrogen. In a nitrogen plasma, the concentration of atomic nitrogen increases as helium or argon partial pressure is increased. After reaching a peak, it decreases with further decrease of nitrogen partial pressure.

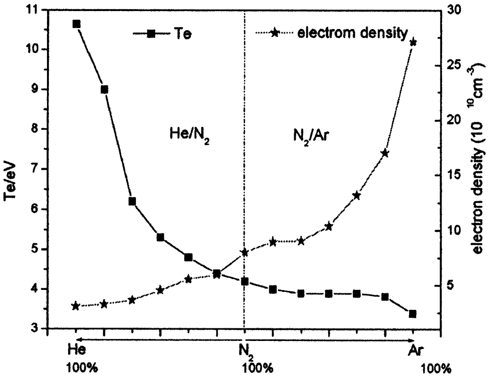

Fig. 2 Electron density and electron temperature vs. gas composition in both N_2/He and N_2/Ar plasmas. The total gas pressure is kept at 0.7 Pa, as measured by a capacitance gauge. The RF power is 480 W.

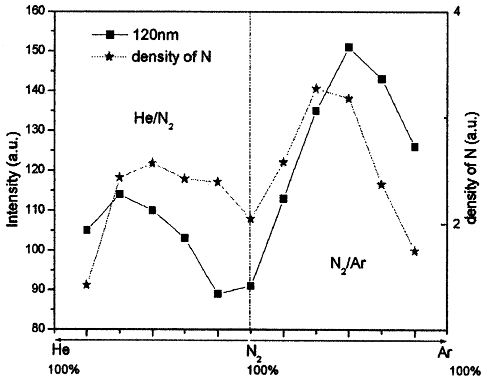

Fig. 3 N I VUV line intensity and line-integrated N I density vs. gas composition in both N_2/He and N_2/Ar plasmas. Plasma parameters are the same as in Fig. 2.

Figure 4 shows the comparison of measured *Te* (by line-ratio technique) and *Ne* in N_2–He mixture discharges in ECR and ICP plasmas. The tuning of *Te* is much more effective in the ICP plasma, since the higher electron density in an ECR plasma leads to a smaller hot-electron tail. We may project that in helicon plasma, where electron density is higher, the effect of *Te* tuning by inert gas mixing would be weaker still.

Self-absorption (radiation trapping), generally strong in VUV wavelength range since most of them are resonance lines, depends on the density, temperature and profile of the absorbing species along the optical path [12]. It was estimated that, due to self-absorption, the measured emission intensity of resonance lines of H and O atoms is reduced by a factor of 10^{-3} or larger in microwave plasma operating at a pressure of 133 Pa or higher [13]. However, at a much lower pressure (less than 0.7 Pa), the absorption mean free path of the emitted photons is estimated to be comparable to chamber dimensions in an ECR etcher [14]. Here, we estimate the effect of self-absorption by measuring and analyzing the shape of radial profiles of VUV emissions under different pressure values.

Radial profiles of VUV emission obtained from helium, argon, and argon–nitrogen mixture plasmas generated in MPS-2000 ICP facility are shown in Figs. 5–7. The asymmetric profile of helium res-

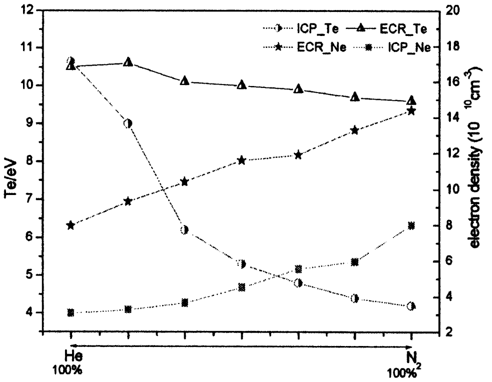

Fig. 4 Electron density and electron temperature vs. gas composition in N_2/He plasmas generated by ECR and ICP reactors. The total gas pressure is fixed at 0.7 Pa, as measured by a capacitance gauge. In order to have the same power density, the RF power is larger (480 W) than the microwave power (100 W) since ECR has a smaller chamber.

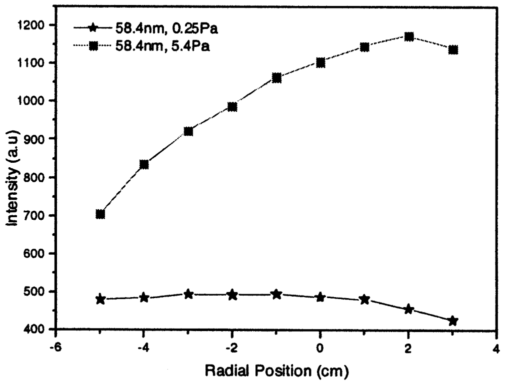

Fig. 5 Radial profiles of VUV emission intensities of He resonance line (58.4 nm) at two different pressures in the MPS-2000 ICP system. The RF power is 480 W.

onance line (58.4 nm) at higher pressure (5.4 Pa) shown in Fig. 5 is an evidence of self-absorption by helium atoms along the optical path. As the mirror is moved closer to the monochromator and hence the absorption length becomes shorter, the measured VUV intensity becomes stronger. At 0.7 Pa or lower, the profile of the above helium resonance line, the argon resonance line at 106.7 nm (Fig. 6), and the atomic nitrogen line at 120.0 nm (Fig. 7) are symmetric, indicating that self-absorption is not important in low-pressure operations.

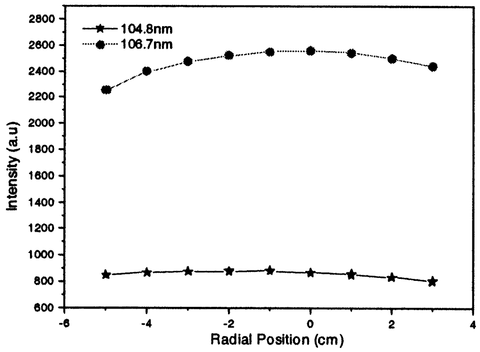

Fig. 6 Radial profiles of VUV emission intensities of Ar resonance lines (104.8 nm and 106.7 nm) in the MPS-2000 ICP system. The gas pressure is 0.6 Pa and RF power is 480 W.

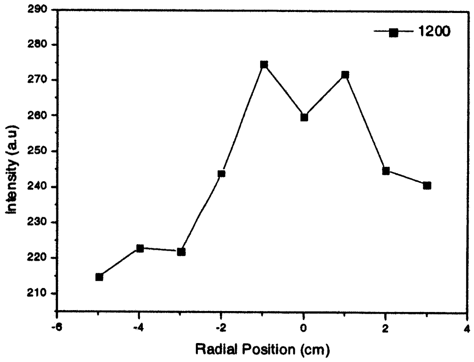

Fig. 7 Radial profile of VUV emission intensities of atomic nitrogen resonance line (120.0 nm) in the MPS-2000 ICP system. The gas pressure is 0.5 Pa and RF power is 480 W.

CONCLUSION

Inert gas mixing is an effective way to change both *Te* and electron density in a low-pressure plasma discharge. It is also an effective technique to enhance the production of certain active species, for example, atomic nitrogen in the case of nitrogen plasmas. However, the higher the electron density, the weaker the *Te* tuning effect due to electron–electron collisions. VUV emission spectroscopy, combined with visible-NIR spectra, are useful tools to determine relative concentration of ground-state active species and its relationship with other plasma parameters.

ACKNOWLEDGMENTS

The authors are grateful for the generous help from Dr. Jim Terry from MIT, Prof. André Bouchoule from University of Orléans, Dr. T. K. Chu from Princeton, and Prof. Long Wang from the Institute of Physics, Chinese Academy of Sciences. This work is partially supported by the National Science Foundation of China.

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