Rare Gas Metastable Atom Density in Diluted O₂ RF Plasmas

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Keywords: metastable atom, oxygen plasma, optical absorption spectroscopy, rare gas dilution

Rare gas diluted O₂ plasma is gaining interests for its advantages in growth rate and film quality of SiOₓ used for ULSI and TFT-LCD. The effect of rare gas dilution has been thought as the enhancement of O atom production via collisional quenching of rare gas metastable atoms electronically excited in plasmas. We have investigated the change of rare gas metastable density due to the increase of O₂ fraction in the radio frequency capacitively coupled plasma.

Rare gas metastable density was measured by optical absorption spectroscopy. The light source is a low pressure discharge lamp of inductively coupled plasma and the probe emission is detected by a photomultiplier with a monochromator. The O atom density in the plasma is also measured by optical absorption spectroscopy in vacuum ultra violet (VUV) range with a VUV monochromator. In order to check the density of energetic electrons, the emission from the higher excited state is monitored. The emission from resonance states adjacent to the metastable state is detected to assure the density change of the metastable.

Under the condition of total pressure of 50 mTorr and 40 W, the O atom density is not significantly reduced even for highly Ar diluted case. As seen in Fig. 1, Ar metastable density decreases with the increase of O₂ fraction in the gas. The emission from resonance states also decreases rapidly with the increase of O₂. In contrast, the emission from the higher excited state (750.38 nm) gradually decreases with the increase of O₂. This indicates the introduction of O₂ into the Ar plasma gives small influence on the density of energetic electrons and subsequently on the production rate of metastable atoms, while Ar metastable is effectively quenched by O₂. This also shows even for small fraction of O₂ in rare gas plasmas, O atoms are selectively produced by rare gas metastable atoms through collisional energy transfer.

The trend is also seen for higher gas pressure of 500 mTorr. Due to the increase of collision frequency, the decrease of metastable appears at lower O₂ fraction. Numerical analysis shows more than 90% of metastable atoms are quenched by O₂ at 3% of O₂ fraction, which corresponds to the real processing conditions.

Figure 2 shows the change of each rare gas metastable density with the increase of O₂ fraction in the plasma. The decrease of each metastables corresponds with the reported quenching rate coefficient. Krypton metastable has the highest density among the four rare gases. This should explain the cause of high oxide growth rate with Kr diluted O₂ plasma in real processing conditions.
Rare Gas Metastable Atom Density in Diluted O2 RF Plasmas

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Rare gas diluted O2 plasmas are gaining interests for application to high quality SiO2 film formation. The density of rare gas metastable atoms and O atom in rare gas diluted O2 radio frequency (RF) capacitively coupled plasma (CCP) was measured by optical absorption spectroscopy (OAS). Decreases of rare gas metastable densities due to addition of O2 indicate efficient O atom production by rare gas metastables via collisional quenching. Krypton metastable had highest density among four rare gas species for fixed RF power. The decrease of Ar metastable density due to O2 addition showed quantitative agreement with reported quenching rate coefficient. Detailed discussion on different gas pressures illustrates reduced O2 fraction is the key for selective production of O atoms through rare gas metastables.

Keywords: metastable atom, oxygen plasma, optical absorption spectroscopy, rare gas dilution

1. Introduction

Non-equilibrium RF plasmas at low pressures are widely applied to various dry processes in semiconductor industry, which are removal and deposition of organic and inorganic materials(1). Major target of oxygen containing plasma has been the removal of photoresist patterns on the substrate (ashing) after the etching process. Formation of thin SiO2 layer for thin film transistors (TFT) of integrated circuits by low temperature processing with oxygen-based plasma is interested(2). Low temperature formation of oxides is also under trial for TFTs of liquid crystal displays (LCD) to reduce the interface state density. Ueno(3) and Sekine(4) showed the enhancement of growth rate of SiO2 films can be achieved by rare gas diluted O2 plasmas. Improvements of film quality using Kr diluted O2 plasma are also reported by both groups. They claim the improvements are due to selective production of oxygen atoms through rare gas metastable atoms. However, the kinetics of rare gas metastable atoms and its relation to the oxygen atom production are not clearly demonstrated.

The measurement of rare gas metastable atom density in plasmas have been performed by several groups. McMillin presented two dimensional profile of Ar metastable density in RF CCP with laser induced fluorescence (LIF)(5). Hebner discussed on two dimensional profiles of density and temperature of Ar metastable atoms in an inductive coupled plasma (ICP) both with laser absorption spectroscopy (LAS) and LIF(6). Makabe also performed two dimensional diagnostics of Ar metastable atom density in ICP with LAS(7).

Here we discuss on the role of rare gas metastables in rare gas diluted O2 plasma based on the density variation of rare gas metastable and O atom due to the change of O2 fraction in the RF CCP using OAS.

2. Experiment

2.1 Diagnostic Methods In this manuscript, we apply four kinds of optical diagnostics, that are the combination of two optical diagnostic methods for two spectral ranges. OAS is used in visible range for the measurement of rare gas metastable density, and in vacuum ultra violet (VUV) range for measurement of O atom density. Optical emission spectroscopy (OES) of excited Ar is used in visible range as a probe of high energy electrons in the plasma while OES of Ar in VUV is a measure of resonant emission which highly correlated to the metastable density.

Figure 1 shows the energy diagram of Ar atom. Ar metastable atom (Ar4s[3/2]) in the plasma is detected by OAS probing the attenuation of 763.51 nm emission (See (a) in Fig. 1) from a discharge light source. Since Ar4s[3/2]) atom is majority among two metastable states of Ar, following data of metastable Ar are focused on Ar4s[3/2]). Ground state O atoms are detected by our vacuum ultra violet absorption spectroscopy (VUVAS)(8) which use emission from O(3P2) to O(1D) at 130.6 nm for obtaining ground state O atom density. With the comparison of two absorption data on rare gas metastables and O atoms, role of rare gas metastables on oxygen dissociation and its extent can be elucidated. Here, we must keep in mind that rare gas metastable density is greatly affected by electron density(9). While the O2 fraction in the gas is increased, electron density should decrease due to electron attachment to O2 (e + O2 → O + O2). This change of electron density affects the production and quenching rate of rare gas metastable and the discussion on the relation of metastable density and O atom production becomes difficult. Therefore, we measured optical emission from excited Ar as seen (b) in Fig. 1. Because excitation rate of Ar4p[1/2]) (ε = 13.5 eV) is almost proportional to that of Ar4s[3/2]) (ε = 11.5 eV), 750.38 nm emission should represent the net excitation rate of metastables(10). With addition of the OES data of excited Ar, the discussion on the relation of rare gas metastable density and O atom production is possible. OES in VUV range of Ar (resonance
emission) is applied for investigating the change of metastable state density which correlate with resonance state density. One of the main loss mechanisms of metastable atoms is the electron collision which lead to nearby resonance or higher electronic excited states\(^{(7)(11)}\). The change of metastable density due to O\(_2\) addition would appear also on the resonance state density and the OES signal from resonance state can give us an idea on the assurance of the absorption measurement.

### 2.2 Apparatus

Figure 2 (a) shows the experimental setup of the optical absorption spectroscopy. CCP reactor has a cylindrical structure with two stainless steel parallel plate electrodes with a gap distance of 30 mm. Diameter of the upper and the lower electrodes are 80 mm and 110 mm respectively, and inner diameter of the reactor is 145 mm. The chamber is evacuated by a turbomolecular pump (TMP) to 1 x 10\(^{-6}\) Torr. O\(_2\) gas diluted by rare gas is introduced from the side of the CCP reactor. Driving frequency of the CCP is 13.56 MHz in this case. Light source chamber attached to the CCP reactor is separated by a MgF\(_2\) window. The light source chamber is evacuated by a rotary pump. Pure rare gas is supplied to the light source chamber and the pressure of the rare gas is normally kept at 10 mTorr. 10 turn coil in the light source chamber is connected to a VHF power supply with 70 MHz of driving frequency via an impedance matching circuit. VHF current on 10 turn coil forms induced field in the chamber and an ICP of rare gas is established. Optical emissions from the rare gas plasma, passing through the CCP, are collected by an optical fiber bundle at the outside of the CCP reactor and guided to a monochromator with a photomultiplier tube (PMT). Output of the PMT is amplified and counted by electronics.

Figure 2 (b) shows the diagram of the VUV emission spectroscopy. 250 mm VUV monochromator, attached to another port of the CCP reactor, is differentially pumped by a TMP. The width of the input and the output slits of the VUV monochromator are set to 0.03 mm. The selected VUV emission from the rare gas resonance state in the CCP is converted to visible range by sodium salicylate located out of the exit slit, detected by PMT, and counted by the same electronics.

### 2.3 Optical Diagnostics

The absorption measurement is performed by a combination of on and off states of the ICP light source and the CCP. As shown in Table 1, four states can be arranged and the signal obtained for each state are named from \(I_1\) to \(I_4\). Absorption rate \(A\) can be obtained by \(A = 1 - (I_2 - I_1)/(I_3 - I_4)\). Pulse modulation of the RF power to main CCP at 10 kHz is applied for the improvement of signal to noise ratio of the absorption measurement\(^{(10)}\).

The relation of the absorption rate and the rare gas metastable density is theoretically expressed as following. Assuming Gaussian line profile of the line shapes of the emission and the absorption, and with the exponential attenuation of the probe emission in the main plasma, absorption rate can be expressed as\(^{(13)}\):

\[
A = \frac{\int \exp \left( -\frac{\omega - \omega_0}{k L T_2} \right) \left[ 1 - \exp \left( -k L e^{-\omega T_2} \right) \right] d\omega}{\int \exp \left( -\frac{\omega - \omega_0}{k L T_2} \right) d\omega}
\]

where \(\omega_0\), \(k\), \(L\), \(T_2\), and \(T_3\) are standard frequency, absorption coefficient, depth of the main plasma, temperature of the atoms in
the light source and that in the main plasma. \( \omega \) is denoted as
\[
\omega = \frac{2\sqrt{\ln(2)(\nu - \nu_d)}}{\Delta \nu_d} \quad \text{.......................................... (2)}
\]
while \( \nu, \nu_d \) and \( \Delta \nu_d \) are real frequency, line center frequency, and the Doppler width. Rare gas metastable atom density \( (n_{m}) \) is related to the absorption coefficient \( k \) as
\[
k = \frac{2}{\Delta \nu_d} \frac{\ln(2)}{8\pi} A_{\nu} g_1 \quad n_{m} \quad \text{.......................................... (3)}
\]
Here, \( A_{\nu}, g_1, g_2 \), and \( g_2 \) are line center wavelength, transition probability, and statistical weights of the lower and the upper state of the transition. The relation of metastable density and the absorption rate of probe emission is shown in Fig. 3 for the case of Ar. From the result of the absorption spectroscopy measurement, we first gain absorption rate \( A \), and applying the value to the relation of Fig.3, we can find absolute metastable density in the plasma. In our configuration, Ar metastable density in the range between \( 3 \times 10^8 \) to \( 1 \times 10^{11} \) can be reasonably evaluated.

Absorption spectroscopy for other rare gas metastable atoms are also possible in the same procedure. Probe emission for each rare gas metastables are 388.86 nm for He \( (2S_{1}\rightarrow2S_{0}) \), 811.29 nm for Kr \( (5s\rightarrow3p_{1}) \), and 823.2 nm for Xe \( (6s\rightarrow3p_{2}) \).

Absorption spectroscopy for ground state O atoms is possible by applying same theory, but experimentally by aligning the VUV monochromator toward the ICP light source\(^{[10]}\). Emission from \( O(2P_{3/2}) \) to \( O(2P_{1/2}) \) at 130.6 nm is selected as a probe for obtaining ground state O atom density. The density ratio between three ground state atoms \( (P_{3/2}, P_{1/2}, P_{2}) \) is deduced based on the Boltzmann relation, assuming the O atom temperature is the same to that of the chamber wall.

3. Results and Discussion

3.1 Oxygen Atom Density

Absolute oxygen atom density in the rare gas diluted O\(_2\) plasma is shown in Fig. 4 as a function of the O\(_2\) fraction in the parent gas. The pressure is 0.1 Torr and the RF power is kept constant at 40 W. In pure O\(_2\) plasma, O atom density is \( 2.5 \times 10^{11} \) cm\(^{-3}\). With the decrease of O\(_2\) fraction, O atom density decreases constantly for helium, while argon and krypton have value comparable to the case of pure O\(_2\) below 30% of O\(_2\) fraction. The decrease of O atom density for helium may be explained by the enhanced diffusion flux of O atoms to the chamber wall in helium ambient. Diffusion coefficient of O atoms in helium is 4 times higher than that in krypton and 2.6 times higher than that in O\(_2\)\(^{[33]}\). However, the decrease of the O density for O\(_2\) fraction lower than 20% is significant even we consider the effect of the diffusion coefficient. So we believe the decrease can be explained only by a decrease of O atom production rate in highly helium diluted conditions. The energy of helium metastable \( (2S_{1/2}) \) (19.8 eV) is higher than the energy required for O\(_2\)\(^{+}\) ion production (12.1 eV). Therefore, Penning ionization: He\(^{+}\) + O\(_2\) \( \rightarrow \) He + O\(_2\)\(^{+}\) is the dominant reaction path of helium metastable and O\(_2\). Then, the decrease of O\(_2\)\(^{+}\) fraction directly affects the O atom density since electron impact dissociation: e + O\(_2\) \( \rightarrow \) O\(_2\)\(^{+}\) + 2e simply decreases with the decrease of O\(_2\) partial pressure. In case of argon and krypton, O atom density is kept comparable even for highly rare gas diluted conditions. This means rapid increase of dissociation ratio of O\(_2\) with the decrease of O\(_2\) fraction. One of the causes of this phenomenon might be the increase of energetic electrons in the plasma due to reduced vibrational excitation of O\(_2\). But authors infer selective O atom production by rare gas metastable would be the main production process for highly rare gas diluted conditions (e.g. Ar(4s\(3P_{2}\)) + O\(_2\) \( \rightarrow \) Ar(4s\(3P_{2}\)) + O\(_2\)\(^{+}\)). Energy of Ar(4s\(3P_{2}\)) and Kr (5s\(3P_{2}\)) are 11.5 and 9.9 eV and both are enough for O\(_2\) dissociation and less than O\(_2\) ionization. If these rare gas metastables are effective for O atom production, metastable density must be affected via deexcitation by O\(_2\) and the metastable density would show significant decrease from the case of pure rare gas plasmas. In the following sections, we show the behavior of the rare gas metastable densities with the introduction of O\(_2\) into the rare gas RF plasmas.

3.2 Ar Metastable Density

Fig.5 shows the O\(_2\) fraction dependence of the Ar metastable density, the resonance emission intensity, and the emission intensity (750.38 nm) from Ar excited state. The plasma is maintained for 50 mTorr and 40 W of RF power. The result is shown only for O\(_2\) fraction from 0 to 20% for the discussion of highly Ar diluted conditions. First, we can see the emission from Ar gradually decreases with the increase of O\(_2\) fraction. 750.38 nm emission of Ar is dominantly produced by direct electron collision excitation of ground state Ar\(^{[13]}\) and stepwise excitation via metastable Ar can be neglected. So we can use the emission intensity as the probe for the density of energetic electrons in the plasma by the form of
\[
I_{\beta} \sim \int_{0}^{\infty} \int_{0}^{\infty} n_\alpha N_{\alpha} \nu \sigma_\alpha(\epsilon) d\sigma - k_4 N_{\beta} n_j \quad \text{.......................................... (4)}
\]
Here, \( I_{\beta} \), \( n_\alpha \), \( N_{\alpha} \), \( \sigma_\alpha(\epsilon) \), \( Q_{\alpha} \) \( \epsilon \), \( n_j \) are emission intensity, electron density, Ar ground state density, electron velocity, electron energy distribution, excitation cross section, and
excitation threshold energy. $k_p$, $N_p$, $n_e$, $N_e$, and $A_e$ are collisional quenching rate coefficient by gas molecule, gas density, and excited state density respectively. In this case of 50 mTorr, quenching term is negligible. In Fig. 5, compared to the pure Ar case, due to the introduction of O$_2$, the emission intensity at 750.38 nm relatively decreases 5, 10, and 27 % for O$_2$ fraction of 3%, 5%, and 10% respectively. This shows $I_p/N_p$ is almost constant below 5% of O$_2$ fraction, and the density of energetic electron is also constant. Hence, we can assume the production rate of Ar metastable in the plasma is almost constant for O$_2$ fraction below 5%.

On the other hand, Ar metastable density in Fig. 5 shows a steep decrease even for 5% of O$_2$ fraction. This means Ar metastables are effectively quenched by O$_2$. Quenching rate coefficient of Ar(4s$^3$P$^0$) by O$_2$ ($k_{q(O_2)}$) is reported by Setser(13) and is 2.1×10^{-10} cm$^3$s$^{-1}$. It has been reported that collisional quenching by electron is the main loss mechanism of Ar metastables in low pressure Ar plasma(78911). Here, we assume electron and O$_2$ are the main quencher of Ar metastable and relate the production and loss rate as

$$n_e(k_{p,n_e} + k_{q(O_2)n_e}) = A_e$$  \hspace{1cm} (5)

where $n_e$, $k_{p,n_e}$, $n_e$, $k_{q(O_2)n_e}$, and $A_e$ are Ar metastable density, electron quenching coefficient, electron density, O$_2$ density, and net excitation rate of Ar metastable. The rapid decrease of the metastable density due to the increase of O$_2$ density in Fig. 5 is clearly explained by the inverse proportion of $n_e$ to $n_{O_2}$ in Eq. (5). Equation (5) is fitted to the metastable density variation in Fig. 5 with the error of 5% and $k_{p,n_e}$ and $A_e$ are found to be 7.4×10^{-11} s$^{-1}$ and 9.9×10^{-11} cm$^3$s$^{-1}$ for the case of pure Ar plasma. Using this equation, we can say that the net quenching rate by O$_2$ exceeds that by electron at O$_2$ fraction of 2% at 50 mTorr. Because quenching reaction between Ar metastable and O$_2$ results in dissociation, it became clear that Ar metastables work for the production of O atoms in highly Ar diluted O$_2$ plasmas.

The emission from the resonance state (104.82 and 106.66 nm) in Fig. 5 also shows rapid decrease due to the increase of O$_2$ fraction. The possible loss processes of the resonance state are the resonance emission, the quenching by O$_2$, and the transfer to the metastable state by low energy electron collisions. First for resonance emission, the radiative lifetime of 106.66 nm emission is 8.4×10^{-9} s, but it experiences radiation trapping by ground state Ar. The number density of the lower state of the emission is 1.5×10^{12} cm$^{-3}$ and 10^7 times higher than the metastable state. The transition probability of the resonance emission is 1.2×10^{-6} s$^{-1}$ and 1.6 times higher than that of 763.51 nm emission. So we can say that the absorption coefficient of the resonance emission is 1.6×10^{7} times higher than the emission used for probing Ar metastable. Therefore, we can estimate the effective lifetime of the resonance emission to be 1.3×10^{-9} s, and the emission rate to be 0.8×10^{-5} s$^{-1}$. Second, the quenching rate coefficient of Ar(4s$^3$P$^0$) resonance state by O$_2$, reported by Setser(13), is 0.09×10^{-10} cm$^3$s$^{-1}$ and the quenching rate at 10% of O$_2$ fraction is 1.3×10^{-3} s$^{-1}$. Third, a strong coupling of Ar(4s$^3$P$^0$) resonance state and Ar(4s$^3$P$^0$) metastable state via low energy electron collisions (Ar(4s$^3$P$^0$) + e $\to$ Ar(4s$^3$P$^0$) + e) is investigated in Ref. 11, and the rate coefficient for the transfer from resonance state to metastable state of Ar is about 3×10^{-7} cm$^3$s$^{-1}$ for effective field of 1 to 10×10^{-6} Vcm$^{-1}$. When we assume the electron density in the plasma to be 3×10^{-9} cm$^{-3}$, transfer rate ($k_{O_2,n_e}$) of 9×10^{-3} s$^{-1}$ is predicted. This transfer rate is highest among the three loss processes of the resonance state although close to the others. Finally, we should conclude that the resonance state atoms are transferred to the metastable atoms by low energy electrons and the metastable atoms are quenched by O$_2$.

3.3 Pressure Dependence The O atom production via collisional quenching of Ar metastables by O$_2$ should be controlled by collision rate of the two species. In Fig. 6, the change of metastable density with O$_2$ fraction is shown between 50 and 500 mTorr. The pressure increase results in higher quenching rate of Ar metastables due to the increase of partial pressure of O$_2$ as in Eq. (5), and the decrease of the metastable density is clearly shown in Fig. 6. The total pressure of 500 mTorr and O$_2$ fraction of 3% is a reasonable condition for rare gas diluted O$_2$ plasma in wafer processing(13), and we can see in Fig. 6 that Ar metastables are completely consumed for the reaction with O$_2$. Under the condition, metastable density deduced from Eq. (5) is 9.1×10^{12} cm$^{-3}$ and it is comparable to 3.5×10^{12} cm$^{-3}$ in the measurement. $k_{q(O_2,n_e)}$ is 13 times larger than $k_{p,n_e}$ for the case, and this indicates most of Ar metastables are quenched by O$_2$. From this fact, we can say that rare gas diluted O$_2$ plasmas utilize rare gas metastables for dissociation of O$_2$ which is the small fraction (-3%) in the gas.

3.4 Rare Gas Species For engineering point of view, to
identify the rare gas species most effective for wafer processing is important and reports showed the best oxide formation rate and quality for Kr diluted O2 plasma.

In Fig. 7, we have summarized the rare gas metastable density as a function of O2 fraction at 50 mTorr for He (2s 2S1/2), Kr (3s 2S1/2), and Xe (6s 2S1/2). The absolute density of the each rare gas metastables are aligned in the order of electron collision excitation cross section for He, Ar, and Kr. Xe metastable density is lower than Kr metastable density probably due to the collisional transfer to the resonance state. He metastable density is significantly small because of its high energy threshold. Every metastable density decreases with the introduction of O2 into the plasma showing quenching reaction by O2. The quenching rate of Kr metastable is slightly lower than that of Ar due to the lower quenching rate coefficient of 1.6 × 10^{-10} cm^3 s^{-1}. Due to the introduction of 3% of O2, the decrease of Kr metastable is 9.1 × 10^7 cm^3 and almost same to the decrease of Ar metastable (8.0 × 10^7 cm^3). However, the decrease of Kr metastable becomes relatively larger for 10% of O2 fraction (Kr : 1.7 × 10^9 cm^3 and Ar : 1.2 × 10^9 cm^3). If we stand on the idea that the decrease of the rare gas metastable density corresponds to the amount of metastables used for O atom production, Kr diluted O2 plasma has the highest ability of O atom production, and this can explain the highest growth rate of oxide. Further analysis under various gas pressures is required to examine the physics in the real processing conditions.

4. Conclusion

In this paper, we presented the effect of O2 fraction on the density of rare gas metastable atoms and O atom in rare gas diluted O2 RF CCP. At total pressure of 50 mTorr, addition of O2 to Ar plasma is insignificant for the density of energetic electrons in the plasma below O2 fraction of 5%. Ar metastable density is greatly reduced due to the collisional quenching by O2. Because the quenching reaction produces O atoms, the dissociation rate of O2 is increased, and O atom density in the plasma is relatively high even for highly Ar diluted conditions. The selective O atom production through rare gas metastables is found for Ar, Kr, and presumably Xe. He metastable is able to produce O2 and showed no effect on O atom production. The comparison on rare gas metastable densities suggested Kr diluted plasma has the highest ability to produce O atoms among four rare gas species. The quenching rate of Ar metastable by O2 increases with the total pressure. At 500 mTorr, the fraction of O2 required for utilizing Ar metastables for O atom production is less than 3%. In the situation, more than 90% of Ar metastables are lost due to the quenching by O2. In this study, we couldn’t find clear evidence which prove the enhancement of oxidation rate compared to the pure O2 plasma. We believe extensive works on O metastables or O2 metastables would let us clarify the source of the benefits of rare gas diluted O2 plasmas.

References

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