ПАРАМЕТРЫ ПЛАЗМЫ И КИНЕТИКА АКТИВНЫХ ЧАСТИЦ В СМЕСИ CF₄+C₄F₈+Ar

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В данной работе обсуждаются взаимосвязи между начальным составом смеси CF₄ + C₄F₈ + Ar, характеристиками газовой фазы и кинетикой гетерогенных процессов в условиях плазмы индукционного разряда пониженного давления. Целью работы являлось исследование зависимости внутренних параметров плазмы (температура электронов, концентрация электронов, энергия ионной бомбардировки) и кинетики активных частиц от соотношения компонентов CF₄/C₄F₈ в плазмообразующей смеси, а также выявление механизмов влияния указанных параметров на такие характеристики «сухого» травления, как скорость травления и селективность. Исследования проводились методами диагностики плазмы двойным зондом Лангмюрра и моделирования плазмы в условиях планарного индукционного плазмохимического реактора. Эксперименты и расчеты проводились при постоянном давлении газа (10 мTorr), вкладываемой мощности (800 Вт) и мощности смещения (150 Вт), при этом отношение CF₄/C₄F₈ варьировалось изменением парциальных скоростей потока этих газов. Было найдено, что замещение CF₄ на C₄F₈ в плазмообразующем газе CF₄ + C₄F₈ + Ar при водит к снижению скорости генерации атомов фтора и плотности потока на обрабатываемую поверхность из-за снижения концентрации в объеме плазмы. Предположено, что рост концентрации и плотности потока ненасыщенных радикалов CF₄ (x=1,2) в смеси с большим содержанием C₄F₈ при близких к постоянным значениях плотности потока энергии ионов (т.е. близкой к постоянной эффективности ионной бомбардировки) способствует снижению эффективной вероятности взаимодействия атомов фтора за счет увеличения толщины фторуглеродной полимерной пленки на обрабатываемой поверхности.

Ключевые слова: CF₄, C₄F₈, скорость реакции, энергия ионов, концентрация, поток, травление, полимеризация
PLASMA PARAMETERS AND ACTIVE SPECIES KINETICS IN CF$_4$+C$_4$F$_8$+Ar GAS MIXTURE

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This work discusses the relationships between the initial composition of the CF$_4$+C$_4$F$_8$+Ar gas mixture, gas-phase characteristics and heterogeneous process kinetics under the condition of low-pressure inductively coupled plasma. The goals were to investigate how the CF$_4$/C$_4$F$_8$ mixing ratio influences internal plasma parameters (electron temperature, electron density and ion bombardment energy) and kinetics of plasma active species as well as to analyze how the changes in above parameters may influence the dry etching characteristics, such as etching rates and selectivities. The investigation was carried out using the combination of plasma diagnostics by double Langmuir probes and 0-dimensional plasma modeling. Both experiments and calculations were carried out at constant gas pressure (10 mTorr), input power (800 W) and bias power (150 W) while the CF$_4$/C$_4$F$_8$ mixing ratio was varied through the partial flow rates for corresponding gases. It was shown that the substitution of CF$_4$ for C$_4$F$_8$ in the CF$_4$+C$_4$F$_8$+Ar feed gas lowers F atom formation rates and causes the decreasing F atom flux to the treated surface due to decreasing their volume density. It was proposed that an increase in the densities and fluxes of unsaturated CF$_x$ (x=1,2) radicals toward C$_4$F$_8$-rich plasmas at the nearly constant ion energy flux (i.e. at the nearly constant efficiency of ion bombardment) causes a decrease in the effective reaction probability for F atoms through the increasing thickness of the fluorocarbon polymer film on the treated surface.

Key words: CF$_4$, C$_4$F$_8$, reaction rate, ion energy, density, flux, etching, polymerization

INTRODUCTION

Fluorocarbon gases are widely used in the microelectronic industry for dry patterning of silicon wafers and dielectric (SiO$_2$, Si$_3$N$_4$) thin films [1,2]. Among these, the CF$_4$ is characterized by the highest F/C ratio and provides the domination of etching over the surface polymerization process under the typical reactive ion etching conditions [3, 4]. The more polymerizing fluorocarbons (C$_2$F$_6$, C$_4$F$_8$, CHF$_3$ and CH$_2$F$_2$) are normally used for the etching processes which require as much as possible SiO$_2$/Si etching selectivity. When both Si and SiO$_2$ etching mechanisms are sufficiently affected by the solid-state diffusion of etchant species through the fluorocarbon polymer film, the etching rates are rather sensitive to the film thickness than to the flux of F atoms coming from bulk plasma. Accordingly, since the thickness of the polymer film on the SiO$_2$ appears to be lower (due to the destruction of polymer in the reactions with surface oxygen atoms [2,5]), the SiO$_2$ etching rate exceeds that for Si. At the same time, together with increasing SiO$_2$/Si etching selectivity, the decrease in absolute Si and SiO$_2$ etching rates as well as an increase in etching residues take place [3-5]. These facts make difficulties for etching process optimization.

One can expect that the reasonable balance between SiO$_2$/Si etching selectivity, absolute etching rates and etching residues may be achieved by the use of two fluorocarbon gases in one gas mixture. In such gas system, the less polymerizing component (for example, CF$_4$) provides the effective generation of etchant species while the more polymerizing component creates the favorable conditions for obtaining high SiO$_2$/Si etching selectivity. From the other side, the mixing of CF$_4$ with other fluorocarbon gas mandatory results in more
complicated reaction scheme, changes plasma parameters as well as influences the formation/decay kinetics and fluxes for all types of plasma active species. Therefore, an understanding of plasma chemistry in the gas systems with two fluorocarbon components is the important tasks for the correct determination of etching mechanisms and optimal choice of gas chemistry for the given etching process.

The goals of current work were 1) to investigate how the CF$_4$/C$_3$F$_8$ mixing ratio in CF$_4$+C$_3$F$_8$+Ar inductively coupled plasma influences internal plasma characteristics (electron temperature, electron density and ion bombardment energy) as well as the kinetics of plasma active species, their densities and fluxes; and 2) to analyze how the changes in above parameters may influence the dry etching characteristics, such as etching rates and selectivities.

EXPERIMENTAL AND MODELING DETAILS

Plasma diagnostics experiments were performed in a planar inductively coupled plasma (ICP) reactor described in our previous works [6,7]. The experiments were performed at a fixed total gas flow rate ($q$ = 60 sccm), gas pressure ($p$ = 10 mTorr), input power ($W$ = 800 W) and bias power ($W_{dc}$ = 150 W). The initial compositions of CF$_4$+C$_3$F$_8$+Ar gas mixture were set by adjusting the flow rates of the corresponding gases. Particularly, the Ar flow rate $q_{Ar}$ was fixed at 20 sccm, so that the fraction of Ar in the feed gas $y_{Ar} = q_{Ar}/q$ was always 33%. The fluorocarbon gases were mixed at various ratios within $q_{CF_4}/q = 40$ sccm while the maximum flow rate for C$_3$F$_8$ did not exceed 15 sccm. Accordingly, the maximum fraction of C$_3$F$_8$ in the CF$_4$+C$_3$F$_8$+Ar gas mixture reached 25%.

Plasma parameters were measured by double Langmuir probe (DLP2000, Plasmart Inc., Korea). The treatment of $I - V$ curves aimed at obtaining electron temperature ($T_e$) and ion saturated current density ($j_+$) was carried out using the software supplied by the equipment manufacturer. The calculations were based on the Johnson & Malter’s double probes theory [8] with the one-Maxwellian approximation for the electron energy distribution function (EEDF). The total positive ion density ($n_+$) was extracted from the measured $j_+$ using the Allen-Boyd-Reynolds (ABR) approximation [9].

In order to obtain the densities of neutral species, we developed a simplified zero-dimensional kinetic model [6, 10, 11] with using the data of $T_e$ and $n_+$ as input parameters. The set of chemical reactions was taken from our previous work [6]. Since the latter provides the detailed discussion on both kinetic schemes and sources of chemical kinetics data, these issues were not the subjects of current study. The model used following assumptions: 1) The electron energy distribution function (EEDF) is close to Maxwellian one. The applicability of Maxwellian EEDFs for the description of the electron-impact kinetics for CF$_4$-based low-pressure ($p < 50$ mTorr) ICPs has been confirmed by the reasonable agreement between the diagnostic results and modeling [11,12]; 2) Under the given set of process conditions, the electronegativity of CF$_4$+C$_3$F$_8$+Ar plasma with more than 30% fraction of electropositive components is low enough to assume $n_- << n_+$. The reasonability of such approach for the CF$_4$- and C$_3$F$_8$-based ICPs was confirmed in several works by both modeling and experiment [6, 12, 13]; 3) The heterogeneous chemistry of atoms and radicals can be described in terms of the conventional first-order recombination kinetics [6,11]; and 4) The temperature of the neutral ground-state species ($T_{gas}$) is independent on the feed gas composition. Since the experimental data on gas temperature were not available in this study, we took $T_{gas} = 600$ K as the typical value for the ICP etching reactors with similar geometry under the close range of experimental conditions [11, 13]. At least, our previous works [6, 7, 11] made with the same set of experimental equipment showed the reasonable agreement between measured and model-predicted plasma parameters for $T_{gas} = 600$ K. Also, the test model runs indicated no principal differences in gas-phase densities of neutral species obtained from the calculations within the $T_{gas}$ uncertainty ~100 K. Such result looks quite reasonable since the dominant decay channels for atoms and radicals under the low pressure conditions are the heterogeneous processes.

For the analysis of heterogeneous chemistry, the fluxes for each kind of neutral species with the volume density $n$ were calculated as $\Gamma \approx 0.25n_{gas}$, where $\nu_T = (8k_B T_{gas}/\pi m)^{1/2}$. The total flux of positive ions was simply evaluated as $\Gamma_+ \approx j_+/e$.

RESULTS AND DISCUSSION

The substitution of CF$_4$ for C$_3$F$_8$ at $p$, $W$ and $W_{dc}$ = const results in slightly increasing $T_e$ (3.7-3.9 eV for 0-25% C$_3$F$_8$), but suppresses the values of both $j_+$ (1.11-0.97 mA/cm$^2$ for 0-25% C$_3$F$_8$) and $n_+ \approx n_e$ (4.5 $10^{10}$-3.8 $10^{10}$ cm$^{-3}$ for 0-25% C$_3$F$_8$). The corresponding data are shown in Fig. 1. The behavior of electron temperature is probably connected with the decrease in electron energy loss for the electronic excitation and ionization of the dominant neutral species. As for the decreasing tendencies for $n_e$ and $j_+$, it may be caused by the combination of following reasons. First, since the addition of C$_3$F$_8$ results in only weak change of $T_e$, the ionization rate coefficients, $k_{i+}$, for all types of neutral species may be assumed to be independent on CF$_4$/C$_3$F$_8$ mixing ratio. According to Ref. [14], the absolute values of $k_{i+}$ for CF$_4$, CF$_2$, CF$_3$ and C$_3$F$_4$, which are the dominant neutral species in CF$_4$
and C$_3$F$_8$ plasmas, are rather close. That is why, one cannot expect an increase in the effective ionization frequency toward increasing C$_3$F$_8$ content in a feed gas. And secondly, under one and the same operating conditions the electronegativity of the C$_3$F$_8$ plasma is lower than that for CF$_4$ one [12,14]. Therefore, the substitution of CF$_4$ for C$_3$F$_8$ in the CF$_4$+C$_3$F$_8$+Ar gas mixture leads to increasing electron diffusion coefficient and thus, to increasing electron decay rate on the reactor walls. Accordingly, the decreasing n$_e$ results in the same behavior of n in order to keep the plasma quasi-neutrality. Physically, the last effect is supported by the decreasing ionization rate.

It was found that, under the given set of operating conditions, the main sources of F atoms in 67% CF$_4$ + 33% Ar plasma are the electron-impact dissociations of CF$_4$ (R1: CF$_4$ + e → CF$_3$ + F + e, R2: CF$_4$ + e → CF$_3$ + F + 2e) and CF$_3$ (R3: CF$_3$ + e → CF$_2$ + F + e). The increasing density of CF$_2$ radicals lifts up the density of CF$_2$ (n$_{CF_2}$ = 1.6·10$^{13}$-3.4·10$^{13}$ cm$^{-3}$, or by ~160 times for 0-25% C$_3$F$_8$) and CF$_2$ (n$_{CF_2}$ = 2.8·10$^{12}$-4.1·10$^{13}$ cm$^{-3}$, or by ~15 times for 0-25% C$_3$F$_8$) radicals. The reason is that the C$_3$F$_4$ directly appears from C$_3$F$_8$ through R9: C$_3$F$_8$ + e → 2CF$_4$ + e while the CF$_2$ is the main dissociation product of C$_3$F$_4$ in R10: CF$_3$ + e → 2CF$_2$ + e. The increasing density of CF$_2$ radicals lifts up the density of CF$_2$ (n$_{CF_2}$ = 1.3·10$^{13}$-2.9·10$^{13}$ cm$^{-3}$, or by ~2 times for 0-25% C$_3$F$_8$) mainly due to the increasing CF$_2$ + F → CF$_3$ recombination rate both in bulk plasma and on reactor walls. As a result, the contributions of R3 and R4 to the total F atom formation rate exceeds the level of R1 and R2 after 20% C$_3$F$_8$ in CF$_4$+C$_3$F$_8$+Ar gas mixture. Thought the rate coefficients for R3 (8.9·10$^{-10}$, 1.1·10$^{-9}$ cm$^3$/s for 0-25% C$_3$F$_8$) and R4 (1.2·10$^{-9}$, 1.6·10$^{-9}$ cm$^3$/s for 0-25% C$_3$F$_8$) exceed the sum of $k_1 + k_2 = 5.8·10^{-10}$-8.3·10$^{-10}$ cm$^3$/s, an increase in C$_3$F$_8$ fraction in a feed gas does no lead to increasing F atom formation rate. The reason is that a weak increase in the F atom formation efficiency toward C$_3$F$_8$-rich plasmas is overcome-pensated by decreasing n$_e$. Simultaneously, the substitution of CF$_4$ for C$_3$F$_8$ accelerated the decay of fluorine atoms through CF$_4$ + F → CF$_3$+ (x = 1, 2) as well as introduces the new effective decay channel in R11: CF$_3$ + F → CF$_2$ + F$_2$ (k$_{11}$ = 4.0·10$^{-11}$ cm$^3$/s). That is why the F atom density decreases monotonically (n$_F$ = 1.6·10$^{-13}$-2.5·10$^{-12}$ cm$^{-3}$, or by ~6 times) toward higher contents of C$_3$F$_8$ in CF$_4$+C$_3$F$_8$+Ar gas mixture.

It was found that under the given set of operating conditions the main sources of F atoms in 67% CF$_4$ + 33% Ar plasma are the electron-impact dissociations of CF$_4$ (R1: CF$_4$ + e → CF$_3$ + F + e, R2: CF$_4$ + e → CF$_3$ + F + 2e) and CF$_3$ (R3: CF$_3$ + e → CF$_2$ + F + e). These processes constitute approximately 85% of the total F atom formation rate while the contribution from the CF$_2$ and CF radicals through R4: CF$_2$ + e → CF + e does not exceed 5% due to the much lower densities of corresponding species. The remaining 10% comes from R7: F$_2$ + e → 2F + e, which is supported by the high F → F$_2$ recombination rate on the reactor walls.
Accordingly, the decay of F atoms is mainly caused by their heterogeneous recombination while the rate of the fastest bulk process $R8$: $CF_3 + F \rightarrow CF_4$ is $\sim 10$ times less.

As can be seen from Fig. 2, the substitution of $CF_4$ for $C_2F_6$ in the $CF_2+CF_4+Ar$ gas mixture results in rapidly increasing densities of both $C_2F_4$ ($n_{CF_4} = 2.1 \cdot 10^{11}, 3.4 \cdot 10^{13}$ cm$^{-3}$, or by $-160$ times for 0-25% $C_2F_6$) and $CF_2$ ($n_{CF_2} = 2.8 \cdot 10^{12}, 4.1 \cdot 10^{13}$ cm$^{-3}$, or by $-15$ times for 0-25% $C_2F_6$) radicals. The reason is that the $C_2F_4$ directly appears from $C_2F_6$ through $R9$: $C_2F_6 + e \rightarrow 2CF_2 + e$ while the $CF_2$ is the main dissociation product of $C_2F_4$ in R10: $C_2F_4 + e \rightarrow 2CF_2 + e$. The increasing density of $CF_2$ radicals lifts up the density of $CF_3$ ($n_{CF_2} = 1.3 \cdot 10^{13}, 2.9 \cdot 10^{13}$ cm$^{-3}$, or by $-2.2$ times for 0-25% $C_2F_6$) mainly due to the increasing $CF_2 + F \rightarrow CF_3$ recombination rate both in bulk plasma and on reactors walls. As a result, the contributions of $R3$ and $R4$ to the total $F$ atom formation rate exceeds the level of $R1$ and $R2$ after 20% $C_2F_6$ in $CF_2+CF_4+Ar$ gas mixture. Though the rate coefficients for $R3$ $(8.9 \cdot 10^{-10}, 1.1 \cdot 10^{-9}$ cm$^3$/s for 0-25% $C_2F_6$) and $R4$ $(1.2 \cdot 10^{-9}, 1.6 \cdot 10^{-9}$ cm$^3$/s for 0-25% $C_2F_6$) exceed the sum of $(k_1 + k_2) = 5.8 \cdot 10^{-10}, 8.3 \cdot 10^{-10}$ cm$^3$/s, an increase in $C_2F_2$ fraction in a feed gas does no lead to increasing $F$ atom formation rate. The reason is that a weak increase in the $F$ atom formation efficiency toward $C_2F_6$-rich plasmas is overcome-pensated by decreasing $n_e$. Simultaneously, the substitution of $CF_4$ for $C_2F_6$ accelerated the decay of fluorine atoms through $CF_4 + F \rightarrow CF_3 + F$ ($k_{1,2} = 4.0 \cdot 10^{-11}$ cm$^3$/s). That is why the $F$ atom density decreases monotonically ($n_F = 1.6 \cdot 10^{13}, 2.5 \cdot 10^{12}$ cm$^{-3}$, or by $-6$ times) toward higher contents of $C_2F_6$ in $CF_2+CF_4+Ar$ gas mixture.

The changes in plasma parameters and composition described above allow one to make some approaches concerning the changes in etching rates, selectivities and etching residues. According to Refs. [6,15], the rate of chemical etching pathway in the fluorine-containing plasmas can be expressed through the flux of $F$ atoms as $y_F \Gamma_F$, where $y_F$ is the effective reaction probability. The data of Tab. 1 indicate that the flux of $F$ atoms follows the behaviors of $n_F$ and show the same decreasing tendency toward $C_2F_6$-rich plasmas ($3.5 \cdot 10^{17}, 5.6 \cdot 10^{16}$ cm$^{-2}$s$^{-1}$ for 0-25% $C_2F_6$). Assuming $y_F = const$ at constant surface temperature, this fact obviously points out on the depletion of the chemical etching pathway. However, the above conclusion is valid only when the etching rate is not limited by the transport of $F$ atoms from the fluorocarbon polymer film. In most of cases, the fluorocarbon polymer film is thick enough to reduce the amount of $F$ atoms on the “film-etched surface” interface compared with that coming from bulk plasma [4,5]. In such situation, the etching kinetics should be described in the terms of the film-thickness-dependent reaction probability which is determined by the balance between polymer deposition and destruction rates [5].
momentum transferred from the incident ion to the surface atom in a single collision \([5]\), one can assume \(Y_s = \sqrt{M_f e_i} \), where \(e_i \approx e \left( -U_f - U_{dc} \right) \) is the ion bombardment energy, \(M_f \) is the effective ion molar mass, \(-U_f \approx 0.5 T_{e}(m_e/2.3m_i) \) is the floating potential and \(-U_{dc} \) is the negative bias provided by \(W_{dc} \). The data of Fig. 1 show that \(-U_{dc} = 209-220 \text{ V} \) for \(0-25\% \text{ C}_4\text{F}_8 \) that results in \(e_i \approx 232-245 \text{ eV} \). The weak changes in \(e_i \) together with \(m_i \) const allow one to apply the linear correlation for \(Y_s = f(e_i) \) and use the parameter \(e_i \Gamma_s \), the so-called ion energy flux, to characterize the rate of the physical etching pathway for the fluorocarbon polymer film. From Tab. 1, it can be seen that the opposite changes of \(e_i \) and \(\Gamma_s \) maintain the ion energy flux on the near-to-constant level up to \(22\% \text{ C}_4\text{F}_8 \) in a feed gas while the overall decrease in \(e_i \Gamma_s \) for \(0-25\% \text{ C}_4\text{F}_8 \) does not exceed \(10\% \). Accordingly, the parameter \(\Gamma_{pol}/e_i \Gamma_s \) characterizing the amount of deposited polymer exhibits the sufficient increase toward \(\text{C}_4\text{F}_8\)-rich plasmas and occupies that range of \(6.0\cdot10^{-20}-7.6\cdot10^{-18} \text{ eV} \cdot \text{cm}^2 \cdot \text{s}^{-1} \) for \(0-25\% \text{ C}_4\text{F}_8 \).

### Table

<table>
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<th>Fractions in a feed gas</th>
<th>(\Gamma_F ), (10^{17} \text{ cm}^2 \cdot \text{s}^{-1} )</th>
<th>(\Gamma_{pol}, 10^{17} \text{ cm}^2 \cdot \text{s}^{-1} )</th>
<th>(e_i \Gamma_s, 10^{18} \text{ eV} \cdot \text{cm}^2 \cdot \text{s}^{-1} )</th>
<th>(\Gamma_{pol}/\Gamma_F )</th>
<th>(\Gamma_{pol}/e_i \Gamma_s, 10^{18} \text{ eV} \cdot \text{cm}^2 \cdot \text{s}^{-1} )</th>
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The results described above allow one to formulate some conclusions concerning the features of the etching kinetics in the given gas system. First, the substitution of \(\text{C}_4\text{F}_8 \) for \(\text{C}_6\text{F}_6 \) in the \(\text{C}_4\text{F}_8+\text{C}_6\text{F}_6+\text{Ar} \) gas mixture suppresses the chemical etching pathway not only due to the decrease in F atom flux, but also through the change in effective reaction probability because of the increasing thickness of the fluorocarbon polymer film on the etched surface. Therefore, one can expect the decreasing absolute etching rates. And secondly, the addition of \(\text{C}_6\text{F}_6 \) probably provides the film-thickness-limited etching regime and thus, creates the favorable conditions for obtaining higher SiO\(_2\)/Si etching selectivity. Accordingly, the reasonable balance between etching rate and selectivity may be effectively adjusted by the \(\text{C}_4\text{F}_8/\text{C}_6\text{F}_6 \) mixing ratio.

### References