ПАРАМЕТРЫ ПЛАЗМЫ И КИНЕТИКА АКТИВНЫХ ЧАСТИЦ В СМЕСИ НBr + Cl₂ + O₂

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В данной работе проведено комбинированное (экспериментальное и теоретическое) исследование характеристик газовой фазы индукционного 13,56 МГц ВЧ-разряда низкого давления в трехкомпонентной смеси HBr + Cl₂ + O₂. Данные по внутренним параметрам плазмы, кинетике плазмохимических процессов и стационарному составу газовой фазы были получены при совместном использовании диагностики плазмы зондами Ланжемора и 0-мерной (глобальной) модели плазмы. Условия эксперимента и моделирования соответствовали постоянным значениям общего давления плазмообразующего газа (p = 10 мтор), вкладываемой мощности (W = 500 Вт), мощности смещения (W_{dc} = 200 Вт) и содержания кислорода в смеси (y(O₂) = 11 %). В качестве варьируемого параметра выступало соотношение начальных концентраций компонентов в паре HBr + Cl₂, которое изменялось в диапазоне 0 – 89 % Cl₂. Было найдено, что замещение HBr на Cl₂ в исследуемом диапазоне условий: 1) приводит к увеличению средней энергии и концентрации электронов; 2) вызывает немонотонное (с максимумом при ~ 45 % Cl₂) изменение концентрации атомов Br; и 3) обеспечивает рост концентрации атомов О в условиях y(O₂) = const. На основе расчетных данных по плотностям потоков активных частиц (атомов Br, Cl и O, положительных ионов) проведен анализ возможных механизмов влияния соотношения начальных концентраций в паре HBr + Cl₂ на кинетику травления Si и SiO₂.

Ключевые слова: параметры плазмы, скорость реакции, поток атомов галогенов, поток энергии ионов, скорости травления Si и SiO₂

PLASMA PARAMETERS AND KINETICS OF ACTIVE SPECIES IN HBr + Cl₂ + O₂ GAS MIXTURE

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In this work, we performed the combined (experimental and model-based) study of gas-phase plasma characteristics for HBr + Cl\textsubscript{2} + O\textsubscript{2} gas mixture under conditions of low-pressure inductive 13.56 MHz discharge. The data on internal plasma parameters, plasma chemistry as well as the steady-state plasma composition were obtained using a combination of Langmuir probe diagnostics and 0-dimensional (global) plasma modeling. Both experimental and modeling procedures were carried out at constant total gas pressure (p = 10 mTorr), input power (W = 500 W), bias power (W\textsubscript{dc} = 200 W) and O\textsubscript{2} fraction in a feed gas (y(O\textsubscript{2}) = 11 \%). The variable parameter was the HBr + Cl\textsubscript{2} mixing ratio, which was changed in the range of 0 – 89 \% Cl\textsubscript{2}. It was found that, under the given set of experimental conditions, the substitution of HBr for Cl\textsubscript{2}: 1) results in increasing both mean electron energy and electron density; 2) causes the mono-monotonic (with a maximum at \sim 45 \% Cl\textsubscript{2}) change in Br atom density; and 3) provides an increase in O atom density at y(O\textsubscript{2}) = const. The possible impacts of HBr + Cl\textsubscript{2} mixing ratio on Si and SiO\textsubscript{2} etching kinetics were estimated through the analysis of model-predicted fluxes for plasma active species (Br, Cl and O atoms, positive ions).

Key words: plasma parameters, reaction rate, halogen atom flux, ion energy flux, Si and SiO\textsubscript{2} etching rates

INTRODUCTION

Silicon and silicon dioxide have found numerous applications in modern electronic device technology [1-3]. Since most of the applications assume precision patterning of the material surface, the development of dry etching processes for these materials is an important task to be solved for achieving advanced device performance.

Until now, there were a large number of experimental and theoretical works dealt with etching characteristics for both Si and SiO\textsubscript{2} in fluorine-based gas chemistries [3–5]. Results of these works provide detailed data on relationships between processing conditions (pressure, input power, and bias power), internal plasma parameters and heterogeneous stages of the etching process. Particularly, it was found that the principal disadvantage of all fluorine-based gas chemistries is the spontaneous Si + F reaction that results in low etching anisotropy of both mono- and poly-silicon. The last problem may be solved by using chlorine- and bromine-based gas chemistries in a form of Cl\textsubscript{2}- and HBr-containing gas mixtures [6]. There were several studies have reported on the plasma-assisted etching characteristics for Si and SiO\textsubscript{2} in HBr- and Cl\textsubscript{2}-based chemistries, for example Refs. [6-12]. When summarizing published data, one should mention following principal issues:

1) The probability of spontaneous chemical reaction Si + Cl/Br at typical process temperatures is much lower than that for Si + F. This effect is normally attributed to bigger sizes of Cl/Br atoms that retard their penetration inside the lattice of etched material. Also, the spontaneous reaction SiO\textsubscript{2} + Cl/Br is thermodynamically forbidden. Accordingly, the steady-state etching of Si and SiO\textsubscript{2} in Cl- or Br-based chemistries requires ion bombardment in order to destruct the Si-O...
bonds and/or sputter the low volatile non-saturated Si-Cl and SiBr₃ compounds.

2) Etching rates of both Si and SiO₂ in HBr plasma are lower than those in Cl₂ plasma under one and the same processing conditions. This fact correlates with differences in volume densities and fluxes of halogen atoms in these gas systems. At the same time, the HBr plasma provides more anisotropies etching together with the better etching selectivity in respect to photoresist masks.

3) The addition of oxygen to HBr or Cl₂ results in more anisotropic etching of Si and SiO₂, but lowers the absolute etching rates for both materials. This effect is attributed to the formation of lower volatile Si-O-Br, and SiO₂Cl₃ compounds that passivate the sidewalls.

Unfortunately, the most of mentioned works had the purely experimental nature and thus, did not analyze the relationships between gas-phase and heterogeneous chemistries. In addition, there are no studies which consider plasma chemistry and etching kinetics in the three-component HBr+Cl₂+O₂ gas mixture. At the same time, the HBr+Cl₂+O₂ gas system may exhibit the positive features of both bromine- and chlorine-based gas chemistries as well as some additional effects due to specific changes in plasma parameters and densities of plasma active species.

In this work, we combined the experimental and model-based approaches to analyze plasma chemistry in HBr+Cl₂+O₂ inductively coupled plasma. The main goals were 1) to determine how the HBr/Cl₂ mixing ratio influences gas-phase plasma characteristics (electron temperature, energy of ion bombardment, densities, and fluxes of plasma active species); and 2) to establish the gas-phase-related parameters applicable for the analysis of Si and SiO₂ etching mechanisms.

EXPERIMENTAL AND MODELING DETAILS

The experiments were performed in the planar inductively coupled plasma (ICP) reactor [13]. The reactor chamber had a cylindrical shape (r = 16 cm, l = 12.8 cm) and was made from anodized aluminum. Plasma was excited using a 13.56 MHz rf power supply connected to a flat copper coil on the top side of the reactor chamber while another 12.56 MHz RF power supply was used to produce the negative dc bias voltage (-Udc) on the bottom electrode. The experiments were conducted at constant gas pressure (p = 10 mTorr), total gas flow rate (q = 45 sccm), input power (W = 500 W) and bias power (Wdc = 200 W). The initial content of each component in the HBr+Cl₂+O₂ gas mixture was adjusted through the corresponding partial flow rates. Particularly, the O₂ flow rate was always 5 sccm that corresponded to y(O₂) = q(O₂)/q = 11%. Accordingly, the variation of q(Cl₂) in the range of 0-40 sccm provided y(Cl₂) = 0-89% as well as the full substitution of HBr for Cl₂.

Plasma parameters were measured by double Langmuir probe tool DLP2000 (Plasmart Inc). The treatment of I-V curves aimed at obtaining electron temperature (Tₑ) and ion saturated current density (jₛ) was based on well-known statements of the double Langmuir probe theory [14, 15]. In order to analyze the chemistry of plasma active species, we developed a simplified zero-dimensional kinetic model with using the data of Tₑ and nₑ as input parameters [13, 16, 17]. The electron density (nₑ) was extracted from the measured nₑ using the solution of the steady-state chemical kinetic equation for negative ions together with the quasi-neutrality equation [16, 17]. Dissociative attachment rate coefficients for dominant electronegative components were either directly taken from Refs. [16-18] or estimated using known process cross-sections [19].

The set of chemical reactions included in the model was taken from a series of published works devoted to the modeling of HBr+Ar [13, 20], Cl₂+Ar [18, 21], O₂+Ar [18, 22], Cl₂+O₂ [18, 23], and HBr+Cl₂ [16] plasmas. The given kinetic schemes (reaction sets with corresponding rate coefficients) have demonstrated an acceptable agreement between model-predicted and measured plasma parameters for HBr, Cl₂ and O₂ [18, 20, 21, 24]. Similarly, to Refs. [13, 16], the model used following assumptions: 1) the electron energy distribution function (EEDF) is close to Maxwellian one; 2) the heterogeneous chemistry of atoms and radicals can be described in terms of the conventional first-order recombination kinetics; and 3) The temperature of the neutral ground-state species (Tgas) is independent on the feed gas composition. Since the experimental data on gas temperature were not available in this study, we took Tgas = 600 K as the typical value for the ICP etching reactors with similar geometry under the close range of experimental conditions [10-12].

For the analysis of heterogeneous chemistry, the fluxes of neutral species with the volume density n were calculated as \( n = 0.25\rho \nu T \), where \( \nu T \) is the thermal velocity corresponding to the given Tgas value. The total flux of positive ions was evaluated as \( \Gamma_e = j_e / e \). The ion bombardment energy was found as \( e_i = -e(U_{dc} + U_i) \), where Udc is the negative dc bias on the bottom electrode provided by Wdc, and \( U_i = 0.5T_{gas} \ln(m_e/2.3m_i) \) is the floating potential. The effective ion mass \( m_e \) was determined using fractions of neutral components with accounting for the differences in corresponding ionization rates and ion transport coefficients.
RESULTS AND DISCUSSION

The principal features of plasma parameters and gas-phase kinetics in the two-component HBr+Cl₂ gas mixture have been discussed in our previous work [16]. Taking into account that the current work 1) deals with processing conditions which are very close to those used in Ref. [16]; and 2) studies the HBr+Cl₂+O₂ system with y(O₂) = const, it would be reasonable to focus the main attention on the effects resulting from the presence of oxygen.

Fig. 1 illustrates the influence of feed gas composition on measured and model-predicted plasma parameters.

Fig. 1. Measured (1, 2) and model-predicted (3–6) plasma parameters as functions of Cl₂ fraction in a feed gas. In Fig. a): 1—electron temperature; 2—ion current density. In Fig. b): 3—total positive ion density; 4—total negative ion density; 5—electron density; 6—relative density of negative ions (n⁻/nₑ). The process condition are: y(O₂) = 11 %, W = 500 W and W₆ ≈ 200 W.

An increase in y(Cl₂) from 0-89% (in fact, the full substitution of HBr for Cl₂ in the HBr + Cl₂ + 11% O₂ gas mixture weak increase in Tₑ (2.5–2.8 eV, see Fig 1(a)) due to the decreasing electron energy loss in inelastic collisions with gas species. The reason is that the higher dissociation degree for Cl₂ molecules) causes a comparison with HBr provides a higher fraction of atomic species in the Cl₂-rich plasmas. Accordingly, since Cl atoms are characterized by higher threshold energies and lower inelastic cross-sections (mainly for electronic excitation and ionization) compared with those for HBr, HCl and Br, the simultaneous increase in y(Cl₂) and decrease in y(HBr) enriches EEDF by the high-energy electrons and shifts Tₑ toward higher values. Similar effects have been reported for many binary gas mixtures, including HBr+Ar, Cl₂+Ar and HBr+Cl₂ plasmas [16, 20, 21]. A growth in both nₑ = 2.5×10⁶ cm⁻³ and nₑ = 2.1×10⁷–7.8×10⁸ cm⁻³ (Fig. 1(b)) is the result of increasing total ionization frequency. This is due to the much higher ionization rate coefficient for Cl₂ molecules (~ 1.3×10⁹ cm³/s vs. ~ 6.7×10⁸ cm³/s for HBr and ~ 6.8×10¹⁰ cm³/s for Br at Tₑ = 3 eV) and an increase in all ionization rate coefficients together with increasing Tₑ (due to εₑ > 3/2Tₑ, where εₑ is the threshold energy for ionization). The behavior of the electron current density (jₑ = 0.5–0.8 mA/cm² for 0-89% Cl₂, see Fig. 1(a)) directly correlates with nₑ. Finally, a decrease in the n⁻/nₑ ratio (10.7-3.5 for 0-89% Cl₂, see Fig. 1(b)) is the result of a nearly constant total negative ion density n⁻ ∼ 2.7×10¹⁰ cm⁻³. The last effect is due to the nearly proportional increase in both total attachment rate and ion-recombination frequency.

Fig. 2 shows the effect of y(Cl₂) on the steady-state densities of neutral species. It was found that, under the given set of process conditions, pure HBr plasma keeps all the features of neutral species kinetics mentioned in previous works [13, 16, 20]. These are as follows: 1) reactions R1: HBr + e → H + Br + e (k₁ ~ 1.6×10⁻¹⁹ cm³/s at Tₑ = 3 eV) and R2: Br₂ + e → 2Br + e (k₂ ~ 1.2×10⁻¹⁰ cm³/s at Tₑ = 3 eV) represent the nearly equal sources of Br atoms; 2) the condition k₁ ≫ k₂ (where R3: H₂ + e → 2H + e) as well as the fast decay of H atoms through R4: H₂ + H → H₂ + Br (k₄ ~ 1.2×10⁻¹¹ cm³/s) and R5: Br₂ + H → HBr + Br (k₅ ~ 1.2×10⁻¹⁰ cm³/s) results in [Br] ≫ [H]; 3) high formation rates for H₂ in gas-phase process R4 and Br₂ in heterogeneous process R6: 2Br → Br₂ (k₆ ~ 125 s⁻¹) lead to [H₂] ≪ [Br₂] ≈ [Br]; and 4) the effective recovery of the original HBr molecules through R5 provides the domination of this species over other neutral components.
The substitution of HBr for Cl₂ in the HBr + Cl₂ + 11% O₂ gas mixture provides the decreasing rates of R1 and R2, but simultaneously introduces new formation pathways for Br atoms through R13: HBr + Cl → HCl + Br (k₁₃ ≈ 2.0×10⁻¹¹ cm³/s) and R14: Br₂ + Cl → Br + BrCl (k₁₄ ≈ 1.5×10⁻¹⁰ cm³/s). Since the latter have quite high rate coefficients, the fast decay of HBr, and Br₂, as well as the rapidly increasing formation rates and densities for both HCl and BrCl are observed. Accordingly, reactions R15: HCl + e → H + Cl + e (k₁₅ ≈ 1.2×10⁻⁹ cm³/s at Tₑ = 3 eV), R16: BrCl + e → Br + Cl + e (k₁₆ ≈ 1.1×10⁻⁸ cm³/s at Tₑ = 3 eV) and R17: BrCl + Cl → Cl₂ + Br (k₁₇ ≈ 1.5×10⁻¹¹ cm³/s) also begin to be an essential source of Br atoms in the range of 20–81% Cl₂. Another important feature is that the rate coefficients for R7–R9 and R10–R12 are noticeably higher than those for R18–R20:

\[
\begin{align*}
\text{Cl}_2 + O & \rightarrow \text{ClO} + Cl \\
(k₁₈ ≈ 3.0×10⁻¹³ cm³/s) & \quad (R18) \\
\text{Cl}_2 + O(¹D) & \rightarrow \text{ClO} + Cl \\
(k₁₉ ≈ 3.6×10⁻¹¹ cm³/s) & \quad (R19) \\
\text{Cl}_2 + \text{OH} & \rightarrow \text{HOCl} + \text{Cl} \\
(k₂₀ ≈ 4.0×10⁻¹⁴ cm³/s) & \quad (R20)
\end{align*}
\]

That is why an increase in y(Cl₂) sufficiently reduces the consumption rates for O, O(¹D) and OH species, provides the rapid increase in their densities (by ~2.7 times for O, ~5.2 times for O(¹D) and ~3.7 times for OH at 0–21% Cl₂, see Fig. 2) and accelerates the dissociation of both HBr and Br₂ through R7–R9 and R10–R12. All these effects, together with increasing electron impact dissociation frequencies for both HBr (k₂₁ = 2.0–9.7 s⁻¹ for 0–89% Cl₂) and Br₂ (k₂₂ = 19–82 s⁻¹ for 0–89% Cl₂), results in non-monotonic behavior for both the effective Br atom formation rate and [Br] value. The density of Cl atoms increase monotonically, but slower than expected from the linear change in y(Cl₂). This is due to the high Cl atom consumption rate through R13, R14 and R17 in HBr-rich plasmas.

The data on plasma parameters and densities of neutral species discusses above allows one to evaluate how the HBr/Cl₂ mixing ratio may influence the Si and SiO₂ etching kinetics. According to Refs. [17, 25], the rate of ion-assisted heterogeneous chemical reactions may be expressed as \( R = \gamma_f \Gamma \), where \( \Gamma \) is the flux of chemically active neutral species, and \( \gamma_f \) is the effective reaction probability. From several published works, it can be understood that 1) the differences in the halogenation degrees for Si surfaces exposed to pure Cl₂ and HBr plasmas are in agreement with the differences in corresponding atom size [26]; 2) the differences in silicon etching rates in pure Cl₂ and HBr plasmas [27, 28] are in agreement with the differences in corresponding halogen atom and ion fluxes [7, 13, 16];...
3) the Si etching yields pure Cl₂ and HBr plasmas at one and the same ion bombardment energy are very close [7]; and 4) the change in Si etching yield with variations of Cl₂/HBr mixing ratio at constant ion bombardment energy does not exceed the experimental error [7]. Therefore, one can suggest the effective reaction probabilities for Br and Cl atoms with Si surface being quite close. Probably, the same suggestion may be applied for SiO₂. As such, one can simply operate with the total chemical active flux ΓBr + ΓCl when analyzing the Si and SiO₂ etching kinetics. The parameter γR for ion-assisted chemical reaction is controlled by several ion-surface interaction pathways [25]. In the case of Si and SiO₂, these are: 1) the ion-stimulated desorption of reaction products, as the boiling points for both SiCl₄ (~ 58 °C) and SiBr₄ (~ 154 °C) [29] exceed the typical process temperature; and 2) the destruction of Si-O bonds, as the corresponding bond strength (~ 800 kJ/mol) is much higher for than for Si-Br (~ 358 kJ/mol) Si-Cl (~ 416 kJ/mol) [29]. The partial rate of each ion-driven pathway is Y₃Γ₊, where Y₃ is the ion-type-averaged process yield. Since Y₃ has a nature of sputtering yield, it is determined by the momentum transferred from the incident ion in a single collision [5]. Accordingly, the influence of HBr/Cl₂ mixing ratio on the kinetics of both ion-stimulated desorption of reaction products and Si-O bond breaking may be characterized by the parameter (Mₑᵢ)₁/₂Γ₊, where Mₑᵢ is the effective ion molar mass.

From Fig. 3, it can be seen that the substitution of HBr for Cl₂ in the HBr+Cl₂+O₂ feed gas results in monotonically increasing ΓBr + ΓCl (6.4×10⁻¹⁶-1.2×10⁻¹⁸ cm²s⁻¹ for 0-89% Cl₂). Also, thought decreases in both -Uₑ₋ (404–351 V for 0-89% Cl₂) and ion bombardment energy (420-368 eV for 0-89% Cl₂) take place, this tendency is completely overcompensated by increasing ion flux (Γ₊ = 2.9×10⁻¹⁵-4.8×10⁻¹⁵ cm²s⁻¹ for 0-89% Cl₂). As such, the parameter (Mₑᵢ)₁/₂Γ₊ also exhibits an increase in the range 4.8×10⁻⁷-6.7×10⁻⁹ eV⁻¹cm²s⁻¹. Though the last fact evidently shows an increase in the ion bombardment efficiency, it does not mean directly the same effect on γR. One can imagine that the behavior γR in HBr+Cl₂+O₂ gas mixture is influenced by an additional factor connected with the chemistry of O atoms. The mechanisms responsible for the negative impact of O atoms on γR for both Si and SiO₂ may be connected with: 1) the formation of Si-O bonds that increases the reaction threshold for F atoms; and 2) the oxidation of reaction products into lower volatile SiBrO₂ and SiCl₂O₂ compounds. Obviously, the last mechanism decreases the desorption yield for reaction products and, thus, lowers the fraction of etched surfaces that are available for the adsorption of F atoms. Assuming that both ion energy flux and the O atoms flux have a nearly proportional, but opposite influences on γR, one can roughly characterize their competitive effect by the Γ₀/(Mₑᵢ)₁/₂Γ₊ ratio. Figure 3 shows that a change in y(Cl₂) results in a much faster increase in Γ₀ (1.1×10⁻¹⁴-1.2×10⁻¹⁶ cm²s⁻¹, or by ~ 110 times for 0-89% Cl₂) compared with (Mₑᵢ)₁/₂Γ₊, and thus, provides a monotonically increasing Γ₀/(Mₑᵢ)₁/₂Γ₊ ratio toward Cl₂-rich plasmas. Therefore, a decrease in γR looks quite probable.

Summarizing above data, one can conclude that the change in HBr/Cl₂ mixing ratio (as well as in any other input parameter which exhibits the similar influence on formation/decay kinetics for plasma active species) may result in the competitive effect of increasing halogen atoms flux and decreasing effective reaction probability. Probably, such situation disturbs

well-known correlations between input process parameters and both Si and SiO\textsubscript{2} etching rates found for F-bases etching chemistries and thus, complicates the optimization of process regimes.

CONCLUSIONS

This work dealt with the effect of HBr/Cl\textsubscript{2} mixing ratio in HBr + Cl\textsubscript{2} + 11% O\textsubscript{2} gas mixture on gas-phase plasma characteristics under conditions of low-pressure inductive 13.56 MHz discharge. The investigation procedure combined plasma diagnostics by Langmuir probes and 0-dimensional (global) plasma modeling. It was found that the substitution of HBr for Cl\textsubscript{2}: 1) results in increasing mean electron energy, electron density and total positive ion density; 2) causes the non-monotonic (with a maximum at ~ 45% Cl\textsubscript{2}) change in Br atom density; and 3) provides the sufficient increase in O atom density. The net changes in gas-phase plasma characteristics may cause the competitive effect of increasing halogen atoms flux and decreasing effective reaction probabilities for both Si and SiO\textsubscript{2}. The most realistic mechanism for the decrease in effective reaction probabilities toward Cl\textsubscript{2}-rich plasmas is connected with the formation of low-volatile SiBr\textsubscript{2} and SiCl\textsubscript{2}O\textsubscript{2} product layers, which reduces the permittivity of halogen atoms and ions toward the etched surface.

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