Characterization of SiO₂ Over Poly-Si Mask Etching in Ar/C₄F₈ Capacitively Coupled Plasma

Received 5 November, 2021; revised 29 November, 2021; accepted 29 November, 2021

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ABSTRACT

This research involved an experimental investigation of the relationship between the plasma parameters and etching properties of SiO₂ over poly-Si mask in Ar/C₄F₈ capacitively coupled plasma (CCP). In these experiments, the etching process was conducted in CCP and the external conditions such as the applied power, pressure, and gas ratio were varied. In addition, the density of radicals, which dominantly participate in surface reactions, the electron density, and the self-bias voltage were measured. As a result, deposition of the CF_x polymer film on the poly-Si mask lowered the electron density and self-bias voltage and the etch rate of the target and the mask increased as the internal parameters of the plasma increased. This result indicated that the electron density and the self-bias voltage, which represent the physical etch elements of ion flux and energy, respectively, have a marked influence on the etching process. Consequently, our work led us to propose a critical value, which is the product of the electron density and self-bias voltage, $n_e V_{bias}$, to analyze the etching mechanism. Our results are also expected to serve as a basic processing database that enables an in-depth understanding of etching.

Keywords: Etching, Fluorocarbon, Ion energy, Ion flux, Plasma

1. Introduction

As the demand for improving the computational capability has been increasing, the feature size and the structure of semiconductors have been shrinking and altered from 2D to 3D-NAND, respectively. In this situation, high-tech etching processes such as atomic layer etching and high aspect ratio etching play a crucial role in creating holes with the required depth. These holes are necessary to produce the desired etch profiles to connect a number of transistors. Creating the desired etch profile requires anisotropic etching and high selectivity and are determined by key plasma parameters such as the electron density, ion energy, and the density of radicals.

Plasma comprising a mixture of argon and fluorocarbon gas is widely used because of its synergistic effect involving both physical and chemical etching. However, it is difficult to clearly characterize the etch properties as the key parameters because of the complexity of the plasma, in which a large number of chemical reactions take place, both in the bulk plasma and on the wafer surface. With respect to etching, not only should the chemical reactions of radicals but also the physical reaction of ions be taken into consideration.

Several previous studies have investigated the effect of changing the etching conditions to analyze the relationship between the etch profile and the internal parameters of the plasma [1–4]. Wendt *et al.* etched the SiO₂ over Si mask using plasma based on a fluorocarbon gas such as C₂H₂F₄ or CHF₃. They reported the etch rate and selectivity by analyzing the chemical factors using optical emission spectrometry (OES) and quadrupole mass spectrometry (QMS) [5]. Oehrlein *et al.* investigated the SiO₂ etching characteristics of C₄F₈/Ar plasma and C₄F₆/Ar plasma by varying the gas ratio and RF bias voltage [6]. In addition to this experimental approach, Kushner *et al.* studied the

kinetics and chemistry of plasma numerically through modeling [7]. Recently, Im *et al.* established a semi-global surface reaction model and suggested the SiO₂ etching mechanism of fluorocarbon plasma [8].

Despite the various studies, it is difficult to obtain the desired etch profile because of the structural differences in the equipment such as the chamber used for the etching process and the complexity of the plasma. Alternatively, a non-global mechanism based on the use of limited parameters could be proposed. Therefore, it is necessary to analyze the etching process by examining the internal parameters of the plasma without reference to specific equipment or external conditions. In this study, the relationship between the internal parameters of the plasma such as the electron density, radical densities, and self-bias voltage on the one hand and the etching characteristics on the other hand, was investigated by conducting SiO₂ etching in Ar/C₄F₈ capacitively coupled plasma under various conditions. This approach enabled us to extract the critical parameter that represents the influence of ions on the etching process, analyze the etching properties when this parameter is used, and roughly divide the etching process into physical and chemical etching regions.

2. Experimental details

The experiments were conducted with a capacitively coupled plasma (CCP) source to which 13.56 MHz RF power was applied. Figure 1 shows a schematic of the CCP source that was employed for processing and diagnosing the SiO₂ over poly-Si mask and determining the plasma parameters, respectively. The CCP chamber has a diameter of 300 mm and contains parallel plate electrodes separated by

Figure 1. Schematic of the chamber and diagnostic equipment set up.

a distance of 50 mm. The top electrode, which includes a shower-head to distribute the gas flow in the chamber uniformly, is grounded and the bottom electrode is connected to the 13.56 MHz RF power generator via an L-type matcher. This system is a highly asymmetric CCP because the grounded electrode includes the grounded wall as well as the top electrode. The wafers used in the etching process were couponed by approximately 15 mm by 5 mm and consisted of a layer of SiO₂ (1,000 nm thick) deposited on a silicon substrate. The SiO₂ layer was masked by a 200-nm-thick poly-Si nanosized hole patterned layer shown in Fig. 2. Here, the contact holes patterned in the mask are 350 nm in diameter and the density of the pattern is different for each position on the wafer; 1:1, 1:1.5, 1:3, 1:5, and 1:10. These different packing densities cause a micro-loading effect in which the reaction area or volume is changed, and thus the density of the etchant near the wafer surface is partially changed and the etching rate is changed. Here, since the wafer for the etching process is couponed in a small size, the effect on the etching can be negligible due to the difference in pattern density. As shown in Fig. 1, these coupon wafers were placed on the bottom electrode in intervals of 7.5 cm starting from the position closest to the nipple. The nipple (42 mm in diameter and 140 mm long) is connected to the chamber to obtain the etching profile and plasma parameters by varying the plasma conditions for constant control parameters. In this experiment, by using a nipple, it is possible to vary the internal parameters of the plasma even under constant external conditions owing to the hollow cathode effect. The steady-state plasma generated in the main chamber and the grounded nipple play the roles of the anode and cathode, respectively. Here, the hot electrons are created by secondary emission resulting from ion bombardment. The electrons accelerate across the plasma potential and are trapped radially within the discharge by the potential across the confined space to build high-density plasma [9]. Thus, the electrons generated by the hollow cathode effect flow into the main chamber with the result that the electron density near the nipple is higher than that at the point at which the electrons exit the nipple 300 mm farther. This causes the radical densities and the ion properties, which represent the chemical and physical reactions, respectively, to change.

The etching profiles were created by using the benchmark external conditions for the RF power and pressure (300 W and 30 mTorr, respectively), and for the Ar:C₄F₈ gas ratio using flow rates of 65 sccm Ar and 6 sccm C₄F₈. The processing time was 10 min. Using these benchmark conditions, experiments were conducted to determine the optimal etch rate and selectivity by changing the above-mentioned external parameters.

The plasma parameters, namely the electron density, radical densities, and self-bias voltage, are well known to play a crucial role in etching a SiO₂ over poly-Si mask. The diagnostics equipment is shown in Fig. 1. The densities of radicals in the bulk plasma were measured using quadrupole mass spectrometry (QMS, RGA200, Stanford Research Systems, USA), by mounting the spectrometer on the chamber sidewall. This spectrometer has a differential vacuum system composed of a rotary pump and turbo molecular pump to maintain the

Figure 2. Schematic of the wafer and definition of etched poly-Si mask and SiO₂ thickness.

base pressure at levels of the order of 10⁻⁷ Torr. The residual gases that are present in the bulk plasma flow into the chamber of the QMS instrument through the 150 orifices located at the end of the 900 mm stainless steel tube to measure the partial pressure at each of the positions. The ions that were ionized from the residual gases by the hot electrons generated by the filament in the ionizer area generate a current signal, which needs to be converted to the radical densities using the method proposed by Singh *et al.* [10]. In this study, the measured radical species were C, F, CF, CF₂, and CF₃ because the other converted radical densities were much lower than those associated with these species. The electron density was measured by a cutoff probe developed by Kim *et al.*, which measures the cutoff frequency in a transmission microwave frequency spectrum (S₂₁). The cutoff frequency, determined as the frequency of the minimum peak ω_c , can indicate the electron density based on the relation of the plasma frequency

$\omega_{pe} = \sqrt{n_e e^2 / \epsilon_0 m_e}$ where n_e is the electron density, e is the electron charge, ϵ_0 is the vacuum dielectric constant, and m_e is the electron mass [11]. The cutoff probe consists of two thin stainless-steel tips that are exposed to the plasma and serve as the microwave transmitter and receiver in the range from 100 kHz to 8.5 GHz, respectively. In the electronegative plasma, the density of the negative ions decreases significantly as the distance between the sheath and the bulk plasma decreases, and eventually approaches zero because the temperature of the negative ions is much lower than the Bohm potential [9]. On the other hand, the change in the electron density from the bulk to the sheath is small compared to the density of the negative ions near the sheath. Because the number of ions cannot exceed the number of electrons to satisfy the quasi-neutral state in the plasma near the electrode surface, the variation in the ion flux escaping from the plasma can be determined by measuring the electron density. The radical densities and electron density at each location are measured by mounting the bellow in the chamber of the QMS instrument. This enables the QMS probe and cutoff probe to be placed inside the main chamber where they are arranged in intervals of 75 mm starting from the position closest to the nipple and extending across the chamber for 300 mm. The ion energy has a significant influence on the sheath voltage waveform, which is the difference between the voltage applied to the bottom electrode and the plasma potential. On the side of the powered electrode, the sheath voltage is dominantly determined by the electrode voltage. This is because the magnitude of the electrode voltage including the self-bias voltage, which is lower between the powered electrode and plasma owing to the difference in the mobility of electrons and ions, is much higher than the plasma potential. This parameter was measured at the bottom electrode with a 100:1 high-voltage probe. The reason why the self-bias voltage was measured at only one point despite the fact that the radical densities and electron density were measured at each point is that the wavelength of the 13.56 MHz RF power is approximately 22 m.

The etching properties, such as the etch rate and selectivity, were measured by cutting the etched coupon wafers into cross-sections and the profiles were acquired using scanning electron microscopy (SEM, TESCAN, Czechia). Using the SEM images of the etched profiles, the

Figure 3. Spatial (a)-(c) radical densities, (d)-(f) electron densities, and (g) self-bias voltage at various power levels: 100, 200, and 300 W at 30 mTorr and Ar:C₄F₈ = 65:6.

Figure 4. (a) Etching profile of the SiO₂ over poly-Si mask and (b) etch rate and selectivity of SiO₂/Si etching at different distances from the nipple and for three different power levels.

etch rate was calculated by dividing the depth of the hole by the processing time. Otherwise, the etched depth of the poly-Si mask was divided by the processing time to obtain the mask etch rate. The selectivity of the target over the mask was calculated by dividing the SiO₂ etch rate by the mask etch rate.

3. Results and discussion

The plasma parameters and etching properties such as the etch rate and selectivity were measured by changing the initial (benchmark) levels of the applied power/pressure/gas ratio from 300 W, 30 mTorr, and

gas flow rates of 65 sccm (Ar) and 6 sccm (C₄F₈), and using a processing time of 10 min. Using the nipple, the plasma uniformity was disrupted to vary the plasma parameters for each location, and the patterned coupon wafers were placed to obtain the etch rate and selectivity. To eliminate the influence of the diagnostic equipment on the etching, the diagnosis and etching were conducted separately.

3.1. Power

Figure 3 shows the radical densities, electron density, and self-bias voltage measured as a function of the applied power and position (distance from the nipple). As shown in Figs. 3(a)–(c), the densities of all considered radical species increases slightly when the RF power or distance increases. In Figs. 3(d)–(f), the electron density increases at higher RF power or at positions closer to the nipple. This can be understood to indicate an increase in the dissociation of the mother gas C₄F₈ as a result of collision with electrons. There are two reasons why the electron density increases with increasing RF power and distance. The first is that the power adsorbed by an electron increases as the RF power increases. The other is the effect of the nipple. The self-bias voltage increases under the same conditions as those under which the densities of the radicals and electrons were measured, as shown in Fig. 3(g). The result of the principle that the self-bias voltage is caused by the difference between the ion and electron mobility shows that the electron density increases with increasing RF power and the electrons trapped at the blocking capacitor increase as the number of electrons escaping from the plasma increases.

Figure 4 shows the etch profiles and etch rate of SiO₂, and the SiO₂ to poly-Si selectivity as a function of the RF power and position (distance from the nipple). As can be seen from Fig. 4(a), the pattern density is different for each position under the same power condition. Although micro-loading effect may occur due to the different pattern density, in the etch rate in Fig. 4(b), the effect is small as it follows the trend of the plasma parameters. The etch rate and selectivity were calculated by dividing the total etched amount and processing time and dividing the etch rates of SiO₂ and poly-Si, respectively. As shown in Fig. 4(b), the etch rate of SiO₂ is the same (100 nm/min) at a distance of 0 cm from the nipple regardless of the RF power (100, 200, or 300 W). However, in close proximity to the nipple, the selectivity cannot be determined because the 1-μm thick SiO₂ is completely etched during the processing time. On the other hand, this shows that the etch rate is

Figure 5. Spatial (a)-(c) radical densities, (d)-(f) electron densities, and (g) self-bias voltage at different pressure levels: 30, 70, and 100 mTorr, applied power of 300 W, and a gas ratio of Ar:C₄F₈ = 65:6.

Figure 6. (a) Etching profile of SiO₂ over poly-Si mask and (b) etch rate and selectivity of SiO₂/Si at different distances from the nipple and at three different pressure levels.

the highest at 0 cm, and it decreases exponentially at distances farther away from the nipple point positioned at 0 cm. In addition, as the applied power increases, all spatial etch rates increase. As is clear from the selectivity in Fig. 4(b), at 0 cm etching does not occur selectively, which is the result of the mask being etched under all conditions. As can be seen in Fig. 4(a), the fluorocarbon layer was deposited on the poly-Si at 150, 225, and 300 mm. According to the definition of the

etch rate, the etch rate of poly-Si becomes negative, and the selectivity of the SiO₂ over poly-Si also becomes negative. Yet, these results and the overall results indicated that the selectivity decreased as the applied power increased.

It is possible to analyze the tendency of the etch properties shown in Fig. 4 with the plasma parameters shown in Fig. 3. The ion flux represented by the electron density was greatly reduced, and the ion energy also decreased owing to the decrease in the self-bias voltage, which ultimately decreased the etch rate. In the case of the selectivity, the tendency is opposite to that of the etch rate, because the C-rich polymer layer on the poly-Si surface increased as the densities of CF and CF₂, which have a relatively low F/C ratio, increased [12].

3.2. Pressure

Figure 5 shows the radical densities, electron density, and self-bias voltage when the pressure was increased from 30 mTorr (benchmarking level) to 100 mTorr. As shown in Figs. 5(a)-(c), the densities of all the considered radical species increased as the pressure increased. In addition, the distribution of radical densities across the chamber became uniform. In the case of the electron density, the effect is less pronounced at distances farther away from the nipple, where the electron density greatly decreased [Figs. 5(d)-(f)]. As the pressure increased, the electron collisions inside the chamber increased, such that the electron density decreased because the electron energy shifted to a lower energy region in which the energy is not sufficient to ionize the neutral gas species.

The variation in the radical densities with increasing pressure can be understood in terms of the tendency of the electron density. As mentioned before in section 2, the electron density can provide an indication of the ion flux. Apart from this, the electron density is well known to be deeply related to the chemical reactions in the bulk plasma [13,14]. Therefore, as the distribution of electrons became uniform, the ionization reactions by electrons in the chamber occurred uniformly, and the distribution of radicals became uniform. As the pressure increased, the amount of C₄F₈, the mother gas in the chamber, increased, and the number of negative ions, C_xF_y, with high electron affinity, also increased. As a result, the electron flux lost to the electrode decreased, and the amount of charge accumulated in the blocking capacitor decreased, thereby slightly lowering the self-bias voltage.

Figure 7. Spatial (a)–(c) radical densities, (d)–(f) electron densities, and (g) self-bias voltage for three different gases ratios: Ar:C₄F₈ = 65:6, 39:18, and 13:30 sccm at 300 W and 30 mTorr.

Figure 6 shows the etch profiles, etch rates, and selectivity as a function of the distance from the nipple and at the three different pressure levels. Referring to Fig. 6(b), at 0 cm, all the SiO₂ had been etched, similar to the experiments in which the applied RF power was varied at 100 nm/min. As shown in Fig. 6(a), at 0 cm, not only the SiO₂ but also the substrate Si is etched. As before, the etching process is not selective at close distance to the nipple, for the above-mentioned reason. As the pressure increased, the densities of all the considered radicals increased (Fig. 5) and those of radicals with a high F/C ratio (such as the F and CF₃ radicals) that form the F-rich polymer layer increased significantly compared to other radicals. Despite the variation in the ion flux and being similar to the variation that was observed by varying the applied power, overetching largely occurred. In comparison with the etch rate vs. power [Fig. 4(b)], the etch rate tended to decrease slightly as the pressure increased [Fig. 6(b)]. With increasing pressure, the electron density decreased slightly. However, the electron density in the chamber, except for those at 0 cm where overetching occurred, maintained a similar order of magnitude and became uniform. Likewise, the radical densities showed the same tendency. In the case of the self-bias voltage, it decreased as the electron density decreased. As a result, the etch rate seemed to decrease, but the change is too small to detect. The selectivity also increased for the same reason that the etch rate increased.

3.3. Gas ratio

Figure 7 shows the results of the measurements of the radical densities, electron density, and self-bias voltage for the Ar:C₄F₈ gas ratios of 65:6, 39:18, and 13:30 under benchmarking conditions.

Figures 7(a)–(c) shows that, as the gas ratio decreases, i.e., as the amount of C₄F₈ gas increases the densities of all considered radical species increases, except for Ar. In addition, the distribution of radical densities in the chamber becomes uniform, for the same reason that the radical species tend to become uniformly distributed with increasing pressure. As the pressure increases, the electron density becomes spatially uniform, and the overall density decreases as the amount of C_xF_y increases with high electron affinity. The self-bias voltage was expected to decrease as the electron density decreases; however, based on the change in the gas ratio, the opposite occurred. Electrons and positive ions were detected to escape toward the chamber wall to match

the quasi-neutrality in the plasma where electron and ion pairs are created by electron-neutral collision. With that, as the C₄F₈ content increased, the number of electrons and positive ions decreased. The decrease in the number of positive ions is much larger than that of electrons and is responsible for the increase in the self-bias voltage as shown in Fig. 7(g). A decrease in the Ar:C₄F₈ gas ratio would lower the ion flux and would tend to increase the densities of all CF_x-based radicals.

Figure 8 shows the etch rate and selectivity for the aforementioned three different Ar:C₄F₈ gas ratios. As shown in Fig. 8(b), the etch rate at 0 cm was the highest. Likewise, the selectivity at 0 cm was not defined because the entire poly-Si mask was completely etched. On the other hand, for the gas ratio of 13:30, the selectivity was 7.3, and all the masks were not etched. This incomplete etching under the specified conditions can be understood by considering that, as the amount of C₄F₈ gas increases, the total amount of CF_x radicals increases. Furthermore, the amount deposited on the mask surface is significantly increased compared with other conditions, which is the result of the formation of a large amount of C-rich polymer as a layer on the surface. Figure 8(b) also shows that the etch rate decreases with the distance from the nipple. As shown in Fig. 7, the densities of radical species CF_x increase, and the ion flux decreases owing to the decrease in the electron density, resulting in an increase in the thickness of the carbon layer, a byproduct of the surface reaction. The self-bias voltage increases by approximately 8%, which has little effect on the etching compared to changes in the radical densities and electron density.

3.4. Critical parameter, $n_e V_{\text{bias}}$

The etch rate and selectivity, which are etching characteristics, are extracted as a synthesis of plasma internal parameters. The etch properties determined by measuring the control parameters could to a large extent depend on structural and environmental differences such as the chamber size, electrode size, and electrode gap. In single-frequency CCP, the ion flux and ion energy must be considered together because it is difficult to independently control these two parameters. Therefore, in this paper, we propose a critical parameter, which is the product of the electron density and self-bias voltage, $n_e V_{\text{bias}}$, and analyzed the etch properties by calculating this quantity. The etch properties were analyzed by conducting an experiment in which we adjusted the

Figure 8. (a) Etching profile of SiO₂ over poly-Si mask and (b) etch rate and selectivity of SiO₂/Si at different distances from the nipple and for three different gas ratios.

parameters to create various conditions in the chamber by breaking down the plasma uniformity using a nipple. In the etching mechanism with plasma, the distinction between physical and chemical etching was ambiguous. We propose that physical and chemical etching can be roughly distinguished by using this critical parameter. The physical etching to be discussed in this paper is etching by ion bombardment, and both mask Si and target SiO₂ are etched. On the other hand, the chemical etching is a process in which two materials are not only etched, but one of them is deposited by chemical effects such as radicals. According to the definition of the etch rate mentioned in Section 2, the etch rate can be obtained by subtracting the post-process thickness from the existing thickness and dividing by the processing time. Using this, it is possible to sufficiently obtain a negative selectivity under certain conditions. For example, in the condition deposited on the mask after the processing, it becomes thicker than the existing mask thickness. This the etch rate of the mask is negative. If the selectivity is calculated using this value, it can be negative.

Figure 9 presents the critical parameter versus the etch rate and selectivity with changes in the control parameters (power, pressure, and gas ratio). In Fig. 9(a), the selectivity is not only divided into negative and positive based on the specific value of the critical parameter, roughly, as the applied power is varied, but the etch rates with negative selectivity could also be considered to be lower than those with positive selectivity. Because the electron density and self-bias voltage can represent the ion flux and ion energy, respectively, the critical parameter, which is the product of these two parameters, represents physical etching. Therefore, Fig. 9(a) shows that it can be divided into physical etching and chemical etching according to the influence of the ions. For variations in the applied power, the etching mechanism is divided based on a critical parameter of approximately $6 \times 10^{12} \text{ V} \cdot \text{cm}^{-3}$, and this also applies to the etch rate and selectivity for changes in the pressure and gas ratio shown in Figs. 9(b) and 9(c). Under these change

Figure 9. Etch rate and selectivity as a function of the critical value, $n_e V_{\text{bias}}$, for different (a) power levels, (b) pressure levels, and (c) gas ratios.

conditions, the regions in which the different etching mechanisms are valid are well differentiated by this specific value of the critical parameter.

4. Conclusions

In this study we examined the relationship between plasma parameters and etch parameters, i.e., the etch rate and selectivity, in the fabrication of semiconductor wafers. A SiO₂ wafer was etched with plasma using a poly-Si nanopatterned mask to analyze the above-mentioned parameters by varying the applied power, pressure, and gas ratio. The plasma analysis involved investigating the electron density, radical densities, and self-bias voltage. The radical density for each species is represented by the chemical properties, and the electron density and self-bias voltage are represented by the ion flux and ion energy, respectively. The results showed that the etch rate rapidly increased and conversely, the selectivity decreased with increasing RF power under the benchmark pressure and gas ratio (30 mTorr and Ar:C₄F₈ = 65:6 sccm), because the electron density and self-bias increased. An increase in the pressure caused a slight decrease in the etch rate and the selectivity increased because the electron density decreased dramatically with a small decrease in the self-bias voltage and the density of the radicals. When the Ar:C₄F₈ gas ratio decreased, the etch rate decreased and the selectivity increased. This is because the

electron density was greatly affected even though the self-bias voltage and etching density increased.

We conducted a fundamental study of SiO₂ over poly-Si mask etching with gas-phase fluorocarbon species, and the results enabled us to propose a new critical value in the form of the product of the electron density and the self-bias voltage. The variation of the etching properties with the critical value, $n_e V_{\text{bias}}$, showed that the mechanism of etching is divided into physical and chemical etching depending on the particular critical value. This study is expected to contribute to the fundamental understanding of etching and serve as a basic processing database.

Acknowledgements

This research was supported by the research fund of Chungnam National University in 2021.

Conflicts of Interest

The authors declare no conflicts of interest.

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