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Selective etching of high-\(k\) HfO\(_2\) films over Si in hydrogen-added fluorocarbon (CF\(_4\)/Ar/H\(_2\) and C\(_4\)F\(_8\)/Ar/H\(_2\)) plasmas

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Inductively coupled hydrogen-added fluorocarbon (CF\(_4\)/Ar/H\(_2\) and C\(_4\)F\(_8\)/Ar/H\(_2\)) plasmas were used to etch HfO\(_2\), which is a promising high-dielectric-constant material for the gate of complementary metal-oxide-semiconductor devices. The etch rates of HfO\(_2\) and Si were drastically changed depending on the additive-H\(_2\) flow rate in C\(_4\)F\(_8\)/Ar/H\(_2\) plasmas. The highly selective etching of HfO\(_2\) over Si was done in the condition with an additive-H\(_2\) flow rate, where the Si surface was covered with the fluorocarbon polymer. The results of x-ray photoelectron spectroscopy indicated that the carbon content of the selectively etched HfO\(_2\) surface was extremely low compared with the preetched surface contaminated by adventitious hydrocarbon in atmosphere. In the gas phase of the C\(_4\)F\(_8\)/Ar/H\(_2\) plasmas, Hf hydrocarbide molecules such as metal-organic compounds and Hf hydrofluoride were detected by a quadrupole mass analyzer. These findings indicate that the fluorine species, carbon, and hydrogen can work to etch HfO\(_2\) and that the carbon species also plays an important role in selective etching of HfO\(_2\) over Si. © 2006 American Vacuum Society. [DOI: 10.1116/1.2187997]

I. INTRODUCTION

As dimensions of metal-oxide-semiconductor field-effect transistor (MOSFET) devices are scaled down in integrated circuits, the gate width will shrink to much less than 100 nm. The thickness of gate dielectrics should be reduced down to 2 nm or less for the present material, SiO\(_2\).\(^1\) Then, the thickness reduction of SiO\(_2\) brings a number of serious problems such as increased gate-leakage current and reduced oxide reliability. Therefore, it will be necessary to integrate the high-dielectric-constant (\(k\)) materials, which can give higher specific capacitance at a larger thickness than SiO\(_2\) and which enable the reduction of gate-leakage current. Integration of high-\(k\) materials will be one of the important issues in scaling MOSFET devices at critical dimensions below 65 nm.

Recently, replacing SiO\(_2\) with silicon oxynitrides of slightly higher dielectric constant has been tried. In the future, high-\(k\) (>20) dielectrics or metal oxides such as HfO\(_2\),\(^2\) ZrO\(_2\),\(^3,4\) HiSi\(_x\)O\(_y\),\(^5,6\) and ZrSi\(_x\)O\(_y\) (Refs. 6 and 7) will be developed to replace SiO\(_2\). When integrating these materials into devices, these materials must be removed completely from the source and drain regions. Therefore, an understanding of the etch characteristics of high-\(k\) materials is required for the removal process.

Plasma etching of high-\(k\) materials has been studied recently for gate dielectric applications. Pelhos et al. reported on the etching of high-\(k\) gate dielectric Zr\(_{1-x}\)Al\(_x\)O\(_y\) thin films with helical-resonator plasmas in Cl\(_2\)/BCl\(_3\).\(^8\) Sha et al. reported on the etching of ZrO\(_2\) with electron-cyclotron-resonance plasmas in Cl\(_2\) and BCl\(_3)/Cl\(_2\).\(^9,10\) Furthermore, Sha et al. also etched HfO\(_2\) thin films in the chlorine chemistries.\(^11,12\) In their studies of HfO\(_2\) etching, chlorine-based chemistries (not fluorine) were chosen because the HfO\(_2\) was prevented from etching in the CHF\(_3\) plasmas where Hf fluoride compound can be formed as the sidewall mask.\(^13,14\) Norasetthekul et al. reported on the etching of HfO\(_2\) with inductively coupled plasmas in Cl\(_2\)/Ar, SF\(_6\)/Ar, and CH\(_2\)/H\(_2\)/Ar.\(^15\) Maeda et al. tried to integrate a MOSFET with a HfO\(_2\) dielectric by using etching in CF\(_4\) and Cl\(_2)/HBr-based chemistries.\(^16\) Emphasis in these studies has been placed on etch chemistries giving the selectivity of more than 1 over the underlying Si substrate and on a better understanding of physics and chemistry for the etching.

The thickness of the gate dielectrics for next-generation MOSFET devices (in the 65-nm technology node and beyond) will be several nanometers. Therefore, selectivity to underlying layers or mask materials will be more important than etch rate in the gate process.\(^17\) From the point of view of HfO\(_2)/Si selectivity, highly selective etching can be achieved in fluorocarbon plasmas. We found that HfO\(_2\) can be etched by fluorine and that the selectivity of HfO\(_2)/Si can be more than 5 in C\(_4\)F\(_8\)/Ar plasmas.\(^18\) In the plasmas, carbon species work as a surface inhibitor on Si not containing oxygen and contribute to obtaining the high selectivity.

To enhance selectivity of HfO\(_2)/Si, HfO\(_2\) etching should be enhanced and/or Si etching should be suppressed. In SiO\(_2)/Si selective etching, fluorocarbon polymer deposited on the surface plays an important role in enhancing the selectivity. The H\(_2\) addition has an effect on the polymer formation in fluorocarbon plasmas.\(^19–21\) Such chemistries may also be applied to HfO\(_2)/Si selective etching. This article presents results of the etching of HfO\(_2\) thin films on Si substrates in inductively coupled hydrogen-added fluorocarbon (CF\(_4\)/Ar/H\(_2\) and C\(_4\)F\(_8\)/Ar/H\(_2\)) plasmas. We discuss the etch
II. EXPERIMENT

The samples for etching were 60-nm-thick HfO2 films on Si substrates prepared by chemical vapor deposition, SiO2 films formed by thermal oxidation, and bare Si. The samples were cleaved into 2-cm² pieces and attached to a 4-in.-diameter Si wafer, which was then clamped onto a wafer stage.

Etching experiments were performed in a low-pressure inductively coupled plasma (ICP) reactor supplied with 13.56-MHz rf power. The reactor consisted of a grounded stainless-steel chamber 25 cm in diameter and 25 cm in height. The rf power was coupled to the plasma via a three-turn planar rf induction coil 15 cm in outer diameter that was positioned on a quartz window 20 cm in diameter and 1.2 cm in thickness located at the top side of the chamber. The wafer stage was 13 cm in diameter and located at the bottom side of the chamber, where a close-fitting ground shield surrounded the stage. The distance from the bottom edge of the rf coupling window to the wafer stage was 5 cm. Gas mixtures of CF4/Ar/H2 plasmas were used, and pure Ar were introduced into the reactor, which was evacuated to a base pressure <1×10⁻⁶ Torr. The gas pressure and flow rate were maintained at 20 mTorr and 5–300 SCCM (SCCM denotes cubic centimeter per minute at STP), respectively.

The discharge was established at a nominal rf power of 280 or 300 W, corresponding to net powers of the r-type matching circuit driving the induction coil. The wafer stage was capacitively coupled to a separate 13.56-MHz rf power supply for additional biasing; the rf bias power was varied between 10 and 150 W (net power), resulting in a dc self-bias voltage on the stage down to between −40 and −160 V.

Sample pieces covered with masks of Si wafer were etched for several minutes. Steps appearing on the sample pieces were measured by stylus profilometry. The chemical composition of the surface was analyzed by x-ray photoelectron spectroscopy (XPS) using Mg Kα x-ray radiation and a pass energy of 50 eV at a takeoff angle of 90°. The plasma parameters (ion density, electron temperature, and plasma potential) were determined by using a cylindrical Langmuir probe located at 2 cm above the wafer stage. The optical emissions from the F atom (3s⁴P⁵/₂−3p⁴D⁵/₂, 685.6 nm), H atom (3p⁴P⁰⁰/₂−3d²D⁵/₂, 656.3 nm), HF molecule (486 nm),23–25 and Ar atom (4s⁴P⁰⁰/₂−4p⁴D⁵/₂, 750.4 nm) were observed to understand the chemical reactions in the gas phase. The etch products were detected by quadrupole mass spectrometry. A commercial quadrupole mass analyzer (QMA) was mounted on the chamber. Gas-phase species were introduced to the differentially pumped analyzer through a 100 μm orifice. The orifice was placed 3 cm from the stage and 2 cm above the wafer surface.

III. RESULTS AND DISCUSSION

A. Selective etching of HfO2 over Si

Figure 1 shows the etch rates of (a) HfO2 and (b) SiO2 in CF4/Ar/H2 plasmas as a function of the additive-H2 flow rate at constant rf powers of 280 W (to the coil) and 50 W (for bias), together with that of Si, and the etch selectivities of (a) HfO2/Si and (b) SiO2/Si. In generating the plasmas, the gas flow rates of CF4 and Ar were 2.5 and 247.5 SCCM, respectively. The pressure was maintained at 20 mTorr. Figure 2 shows the etch rates of (a) HfO2 and (b) SiO2 in CF4/Ar/H2 plasmas. The gas flow rate of CF4 was 2.5 SCCM. The other parameters were the same as in the CF4/Ar/H2 plasmas. The etch depth was measured as a function of etch time up to several minutes and exhibited an approximately linear increase with time. Thus, the etch rate was calculated as the ratio of the depth to time. In the figure, error bars correspond to variance in the measurements, which are not extended to calculating selectivities.

In the CF4/Ar/H2 plasmas, the etch rates of HfO2/Si and SiO2 were maintained to be almost constant in all the tested H2 flow rates. On the other hand, the etch rates of HfO2 and Si in the CF4/Ar/H2 plasmas were drastically changed depending on H2 flow rate. The fluorocarbon polymer was deposited on the Si surface between 4 and 8 SCCM in the H2 flow rate. When H2 is added to fluorocarbon plasmas, fluorine is scavenged by hydrogen with the production of the HF molecule in the gas phase.26,27

![Fig. 1. Etch rates of (a) HfO2 and (b) SiO2 in CF4/Ar/H2 plasmas plotted with that of Si, and etch selectivities of (a) HfO2/Si and (b) SiO2/Si as a function of additive-H2 flow rate. The rf bias power was constant at 50 W.](image-url)
Involving these reactions, the C/F ratio in fluorocarbon species becomes higher. The carbon-rich species are likely to have high sticking probability and to deposit on the surface. If the flow rate of CF$_4$ is increased, much more carbon-rich species are produced. Even in CF$_4$/Ar/H$_2$ plasmas, the species abundant in the gas phase can form deposited films.

At 6 SCCM in H$_2$ flow rate, the polymer also appeared on the HfO$_2$ surface in the C$_4$F$_8$/Ar/H$_2$ plasmas. At 4 and 8 SCCM, however, HfO$_2$ was etched selectively. In the case of a constant rf bias power, the self-bias voltage on the wafer stage was varied from –40 to –70 V with increasing additive-H$_2$ flow rate. The polymer formation on the surface can be affected by ion-bombarding energy changed with the self-bias voltage. Therefore, the etch characteristics were examined in the C$_4$F$_8$/Ar/H$_2$ plasmas at a constant self-bias voltage.

Figure 3 shows the etch rates in the plasmas where the self-bias voltage was maintained at a constant value of –90 V. The etch rates of HfO$_2$, Si, and SiO$_2$ were drastically changed between 0 and 6 SCCM in the H$_2$ flow rate and remained almost unchanged at flow rates more than 6 SCCM. The HfO$_2$ was etched selectively at 2 and 6 SCCM in the H$_2$ flow rate. Since the ion density was decreased with increasing H$_2$ flow rate from 0 to 6 SCCM (Fig. 4), the ion flux to the surface also decreased. The etching reactions of Si and fluorocarbon polymer on the surfaces seemed to be suppressed with decrease of the ion flux.

Optical-emission intensities of F, H, and Ar atoms and HF molecules were measured. It may be crucial to estimate the density of emitting species from the emission intensities. The intensity depends on many factors, such as electron density, electron energy-distribution function, density of the emitting species, excitation cross section of the excited state, and so on. Usually, actinometry is employed to quantify the density.
of species by using noble gases. In this work, all the optical-emission intensities were normalized by the emission intensity of Ar atoms at 750.4 nm. Figure 5 shows the normalized intensities of F and H atoms and HF molecules. Strictly speaking, since there is a difference in the electron excitation cross sections between the target species F, H, and Ar atoms and HF molecules, the normalized intensities cannot represent quantitatively the density of the species. However, since the electron temperature ranging between 2.8 and 3.0 eV in Fig. 4 was not changed significantly over the tested regime, the normalized intensities represent the qualitative trend in density for the species.

The density of H atoms increased with increasing H2 flow rate. Especially, the density increased immediately between 6 and 8 SCCM in H2 flow rate, where the reaction on the Si surface changed from deposition to etching. The F atoms were scavenged from fluorocarbon species and in the gas phase by H atoms from 2 to 6 SCCM in H2 flow rate. The scavenging reaction reached saturation at 6 SCCM. The density of HF molecules increased with the increase of H atoms between 8 and 16 SCCM in the H2 flow rate, where the H atom is abundant.

The density of F atoms between 2 and 6 SCCM in the H2 flow rate became lower than that at 0 SCCM, since H atoms scavenged F atoms with the formation of HF molecules. Then, the density of F atoms was recovered between 8 and 16 SCCM, indicating that F atoms were produced by the electron-impact dissociation of HF molecules. Thus, the etch rate of Si was decreased between 0 and 6 SCCM in the H2 flow rate, with decreasing density of F atoms being an etchant for Si. In this regime, the deposition species, which were produced by the scavenging reaction of F, also reduced the etch rate of Si. The density of SiO2 was maintained, which can be etched by fluorocarbon species (including deposition species on Si surface) as well as by F atoms. Regarding the dissociation reaction of HF molecules, there are two possible paths:

$$\text{HF} + e \rightarrow \text{H} + \text{F} + e,$$

$$\text{H} + \text{HF}^* \rightarrow \text{H}_2 + \text{F}.$$}

Here we consider the role of H atoms in fluorocarbon plasmas. To elucidate the reaction of H atoms, the etch rates of HfO2, Si, and SiO2 were measured in C4F8/H2 and CF4/H2 plasmas (shown in Figs. 6 and 7, respectively). The flow rates of C4F8 and CF4 were 5 SCCM and that of H2 was varied from 0 to 20 SCCM. The power to the coil and self-bias voltage were maintained at 280 W and −90 V, respectively. The pressure was set at 20 mTorr. In the C4F8/H2 plasmas, the etching reaction occurred between 0% and 150% in the gas-mixture ratio of H2/C4F8. The deposition film appeared at more than 150%. However, in CF4/H2 plasmas, the surface reactions on HfO2 and Si were changed from etching to deposition with increasing gas-mixture ratio. Then the reactions were turned into etching again, since the...
deposition film was made thinner with H₂ addition. Furthermore, when the C₂F₆ flow rate is less than 5 SCCM, the thickness of the deposition film is thinner and the etching reaction can occur at more than 400% of the gas-mixture ratio. Therefore, it is essential that the reaction on the Si surface changes from etching to deposition and from deposition to etching with the addition of H₂ atoms. These facts show that the excess H atoms, which do not contribute to the production of HF molecules or are produced by the dissociation of HF molecules, can etch the deposition film of the fluorocarbon polymer. One can understand the etching of fluorocarbon polymer by H atoms by the analogy of the etching of graphite by H atoms in the process of diamond synthesis. In Fig. 3, the etching of HfO₂ and Si can proceed from 8 to 16 SCCM in the H₂ flow rate, since etchants (including F atoms) can reach the HfO₂, Si, and SiO₂ surfaces, as a result of the etching of excess H atoms and removing of the deposition film of the fluorocarbon polymer.

Figure 8 shows the XPS spectra of F₁s and C₁s on HfO₂, Si, and SiO₂ surfaces etched in the C₄F₈/Ar/H₂ plasmas and on preetched surfaces (indicated by notation of “Ref.”). The experimental conditions were the same as in Fig. 3. The values of additive-H₂ flow rate from 0 to 6 SCCM are shown in the graphs.

To understand species reactive with HfO₂, the etch characteristics of HfO₂ were examined in C₄F₈/Ar/H₂ (2.5/247.5/16 SCCM), pure Ar (250 SCCM), and CH₄/Ar (12.5/237.5 SCCM) plasmas. Figure 9 shows the etch rates in these plasmas. Here, the ion energy was defined by \( V_p - V_{dc} \), where \( V_p \) and \( V_{dc} \) correspond to plasma potential and self-bias voltage, respectively. The power to the coil and pressure were maintained at 300 W and 20 mTorr, respectively. The ion densities in the C₄F₈/Ar/H₂, pure Ar, and CH₄/Ar plasmas were \( 1.5 \times 10^{11} \), \( 3.6 \times 10^{11} \), and \( 1.5 \times 10^{11} \) cm⁻³, respectively. The etch rates in the pure Ar plasmas did not exceed those in the C₄F₈/Ar/H₂ plasmas, although the ion density in the pure Ar plasmas was two times higher than that in the C₄F₈/Ar/H₂ plasmas. Therefore, the etching of HfO₂ can proceed with involving chemical reactions related to C, F, and H species in the C₄F₈/Ar/H₂ plasmas. In addition, the etch rates in the CH₄/Ar plasmas did not exceed those in the pure Ar plasmas and is not dependent on the CH₄ flow rate. The deposition of carbon species suppressed etching of HfO₂.

**B. Volatile products in HfO₂ etching**

As mentioned above, at least the F species is necessary to etch HfO₂. The carbon species may also play a role in the etching of HfO₂, as implied by the results of XPS measurements. Understanding the etch mechanism is one of the most important issues in knowing the etchants of HfO₂ in fluorocarbon plasmas. In this study, a QMA with a mass range from 0.4 to 500 amu was used to observe the ionic species.
and the etch products in the gas phase. The ionic species were detected in the $C_4F_8/Ar$ plasmas, where the $C_4F_8$ flow rate, pressure, and power to the coil were maintained at 2.5 SCCM (1% of the total), 20 mTorr, and 280 W, respectively (Fig. 10). The various ionic species of fluorocarbon were observed in the plasmas, including those with mass higher than the parent molecule (>200 amu). In the etching of HfO$_2$ with a self-bias voltage of −90 V, the ionic species as etch products were detected. The several peaks appearing in etching were assigned to $Hf^+$, $HfF^+$, $HF_2^+$, and $HF_3^+$ in the spectrum (Fig. 10), compared with the calculated mass patterns using the relative abundance of naturally occurring Hf isotopes, i.e., $^{177}\text{Hf}$ (18.6%), $^{178}\text{Hf}$ (27.3%), $^{179}\text{Hf}$ (13.6%), and $^{180}\text{Hf}$ (35.1%). These Hf fluoride ions may be produced by the electron-impact dissociation of HfF$_4$. These cannot be identified to be the primary etch products, nor secondary or higher. It is certain that the Hf fluoride must be included in the etch products as volatile species.

In $C_4F_8/Ar/H_2$ plasmas, the etch products were also observed. The gas flow rates of $C_4F_8$, Ar, and $H_2$ were 2.5, 247.5, and 8 SCCM, respectively. The pressure, power to the coil, and self-bias voltage were set at 20 mTorr, 280 W, and −90 V, respectively. These experimental parameters correspond to the condition where HfO$_2$ can be selectively etched (Fig. 3) and where the etch products may contain C atoms, as implied by the XPS results (Fig. 8). Figure 11 shows the mass spectrum of ionic species with mass ranging from 189 to 201 amu. The spectrum was obtained by subtracting the spectrum in the nonbiased condition from that in the with biased condition where the HfO$_2$ could be selectively etched.

The $HfCH_4^+$ and $HfH_2F^+$ may be produced by the dissociation of molecules with the structure like metal-organic compounds. Although $HfC^+$ was detected, the molecule may be produced from $HfCH_4^+$, since the yield of C atoms is too low to etch HfO$_2$. It may be natural that HfO$_2$ is etched with the production of metal-organic compounds as etch products, since such compounds are used for chemical vapor deposition of HfO$_2$. In addition, it may be possible to etch HfO$_2$ by CH$_4$ chemistries with optimized experimental parameters, although the results in CH$_4/Ar$ plasmas (Fig. 9) could not prove this possibility in the present work.

**IV. CONCLUSION**

In the present study, the etch characteristics of HfO$_2$ were examined in CH$_4/Ar/H_2$ and $C_4F_8/Ar/H_2$ plasmas. When $H_2$ was added to the $C_4F_8/Ar$ plasmas, the highly selective etching of HfO$_2$ over Si could be done. The HfO$_2$ was etched even in the condition where fluorocarbon polymer film was deposited on a Si surface.

On the HfO$_2$ surface etched selectively in the $C_4F_8/Ar/H_2$ plasmas, the carbon content was lower than the adventitious hydrocarbon of the atmospheric contaminant on the preetched surface. This implied that the carbon and/or hydrocarbon species may be etchants of HfO$_2$ and the etch products may contain Hf and carbon atoms. The sputtering rates of HfO$_2$ in pure Ar plasmas did not exceed the etch rates in the $C_4F_8/Ar/H_2$ plasmas and were higher than the etch rates in CH$_4/Ar$ plasmas. Therefore, fluorine species are necessary to etch HfO$_2$ in our examples. In the gas phase, $HfCH_4^+$ ($\lambda=0–4$) and $HfH_2F^+$ ($\lambda=0–2$) were detected by QMA. It is demonstrated that metal-organic compound was determined to be one of the volatile etch products in the $C_4F_8/Ar/H_2$ plasmas. The formation of metal-organic compounds is an interesting topic for the etching and depositing of materials containing transition metals. Further analyses will be important for the materials introduced to next-generation devices.
In the actual gate processes, the highly selective etching of HfO$_2$ over Si can be performed with precise control of H$_2$ addition in C$_2$F$_6$/Ar/H$_2$ plasmas. The polymer deposition brought by H$_2$ addition is effective for reducing the etch rate of Si. Furthermore, the polymer may even prevent etching of HfO$_2$ in narrow trenches of musks. Therefore, for practical use, the experimental parameters such as additive-H$_2$ flow rate should be optimized while observing the etch profiles.

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