Selective Etching of Silicon Nitride Over Silicon Oxide using ClF_3/H_2 Remote Plasma

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Abstract

Precise and selective removal of silicon nitride in a $\text{SiN}_x/\text{SiO}_y$ stack is crucial for a current 3D-NAND (not and) fabrication process. In this study, fast and ultra-high selective isotropic etching of SiN_x have been studied using a ClF_3/H_2 remote plasma in an inductively coupled plasma system and a mechanism of SiN_x etching was investigated by focusing on the role of Cl, F, and H radicals in the plasma. The SiN_x etch rate over 800 Å/min with the etch selectivity of ~ 130 could be observed under a ClF_3 remote plasma at a room temperature. Furthermore, compromising the etch rate of SiN_x by adding H_2 to the ClF_3 plasma, the etch selectivity of SiN_x over SiO_y close to ~ 200 could be obtained. The etch characteristics of SiN_x and SiO_y with increasing the process temperature demonstrated the higher activation energy of SiO_y compared to that of SiN_x with ClF_3 plasma.

1. Introduction

As the semiconductor device size is decreased to sub-nanoscale and the device integration is changed from two dimensional to three dimensional structure, more precise and selective process technology is required for the semiconductor device fabrication[1]. In the various semiconductor devices, silicon nitride has been widely used as a barrier layer for dopant diffusion, a gate sidewall spacer layer, a buffer layer, etc. due to high insulating characteristics, high thermal and mechanical stability, etc. and selective etching of silicon nitride over silicon and/or silicon oxide is important for various microelectronic applications[2].

These days, in the three dimensional Not-AND (3D NAND) device fabrication, the number of silicon nitride/silicon oxide ($\text{SiN}_x/\text{SiO}_y$) stack is increasing and the thickness of one $\text{SiN}_x/\text{SiO}_y$ layer is decreasing continuously for higher memory density in the vertical direction. Therefore, the etching of SiN_x layers uniformly and ultra-high selectively to SiO_y layers in the $\text{SiN}_x/\text{SiO}_y$ stack is becoming more challenging process. Until now, the selective etching of SiN_x in $\text{SiN}_x/\text{SiO}_y$ stacks is achieved by wet etching using a hot phosphoric acid (H_3PO_4)[3–6]. In case of the wet etching, however, the penetration of an etch solution into holes is getting more challenging as the thickness of the $\text{SiN}_x/\text{SiO}_y$ layer is decreased and the remaining SiO_y layers can be collapsed due to the surface tension. Moreover, several additives for increasing the etch selectivity of $\text{SiN}_x/\text{SiO}_y$ are found to cause oxide regrowth problems after etching unless its process condition is not carefully controlled[6]. To solve these problems, a dry process for isotropic and selective etching of SiN_x needs to be developed as an alternative technology for 3D NAND device fabrication.

Various studies have been reported for selective etching of SiN_x over SiO_y using dry etch processes. For example, an ultra-high selective etching of SiN_x over SiO_y was reported using CF_4 -based ($\text{CF}_4/\text{O}_2/\text{N}_2$, $\text{CF}_4/\text{CH}_4/\text{Ar}$) gases with a microwave chemical downstream etcher and an inductively coupled plasma (ICP) etcher[7–9]. In addition, NF_3 -based ($\text{NF}_3/\text{O}_2/\text{NH}_3$, $\text{NF}_3/\text{O}_2/\text{N}_2$) gases was used to ultra-high selective

etching of silicon nitride over silicon oxide with downstream etchers based on inductively coupled plasma (ICP) or capacitively coupled plasma (CCP)[9–13]. However, the use of carbon-containing etch gases without adequate additive gases may result in a carbon-contamination or deposition of CH_x polymer on the surface of silicon nitride. Also, due to the high global warming potentials (GWPs) of CF_4 - and NF_3 -based etch gases [GWP values; CF_4 (7,390), NF_3 (17,200)], alternative etch gases need to be studied for environmental aspects in the near future[14].

ClF_3 with the GWP of ~ 0 have been used primarily as an in-situ cleaning gas for chemical vapor deposition (CVD) chambers in replacement of perfluorocarbon compounds (PFC), which have high GWP values or as an etch gas for silicon etching by heating, neutral cluster beam etching, reactive ion beam etching, etc. [15–19]. In this study, ClF_3 remote plasma was applied for a fast and ultra-high selective etching of silicon nitride (SiN_x) over silicon oxide (SiO_y). The etching of SiN_x using ClF_3 showed high etch rate over 800 Å/min and the etch selectivity of SiN_x over SiO_y of ~ 130 . The etch selectivity of SiN_x was further increased with H_2 addition in the ClF_3 plasma. The effect of Cl, F, and H radicals on the selective etching of SiN_x was investigated using plasma and surface analysis tools, and its etch mechanism was suggested.

2. Experimental Section

2.1. Etching of silicon nitride.

Figure 1 is a schematic drawing of a remote type inductively coupled plasma (ICP) etching system used in this study. 13.56 MHz RF power was applied to the planar type ICP coil at upper side of a chamber. For the isotropic etching of SiN_x , double grids with multiple holes with 1.5 mm radius were arranged at the center of ICP reactor to prevent an ion bombardment effect and deliver radicals on the substrate. The substrate temperature was adjusted from 25 to 500 °C by a silicon carbide (SiC) heater connected to an external power supply. The chlorine trifluoride (ClF_3 , 200 sccm) and Argon (Ar, 200 sccm) were flown through a circular shape gas distributor to the process chamber and the operating pressure was maintained at 200 mTorr.

2.2. Sample preparation.

Blank 1.5 μm thick SiN_x thin films and blank 300 nm thick SiO_y thin films, and multilayer stacks composed of repeating SiO_y (270 Å) and SiN_x (270 Å) thin films deposited by a plasma enhanced chemical vapor deposition (PECVD) process (supplied by WONIK IPS Inc.) were used in this experiment.

2.3. Characterization.

The etch rate of SiN_x and SiO_y were measured by a step profilometer (Tencor, Alpha-step 500) and with Scanning Emission Microscopy (SEM, Hitachi S-4700) after patterning with photoresist (PR, AZ 5214E) as an etch mask. Also, the etch profiles of the multilayer thin films composed of $\text{SiN}_x/\text{SiO}_y$ stacks were

observed by the SEM. The surface roughness of films after the etching was measured by atomic force microscope (AFM, XE-100, Park System) with a non-contact measurement mode. The characteristics of ClF_3/H_2 plasma were analyzed with Optical Emission Spectrometry (OES, Avaspec-3648). Byproduct gases during etching process were monitored with Fourier-Transform Infrared Spectroscopy (FT-IR, MIDAC 12000). The binding state and atomic composition of SiN_x and SiO_y (thin films of initial thickness of 5000, 3000 Å, respectively) before and after the etching were analyzed by X-ray Photoelectron Spectroscopy (XPS, MXP10, ThermoFisher Scientific) with a monochromated Al K α source (1,486.6 eV) with spot size of 400 μm . The expected energy resolution of XPS is below 0.5 eV FWHM. The Advantage 5.0 software was used for curve fittings and the areas of each peak were calculated with Shirley background. The incident angle of X-ray to the sample was 50° and a hemispherical sector energy analyzer was positioned perpendicular to the sample stage.

3. Results And Discussion

Figure 2 shows etch characteristics of SiN_x and SiO_y with ClF_3 gas only and ClF_3 remote plasmas. For ClF_3 remote plasmas, 200 sccm of Ar was added to 200 sccm of ClF_3 for the plasma stability. As shown in figure 2a), the etch rates of SiN_x and SiO_y were increased gradually with increasing rf power due to the enhanced dissociation of ClF_3 reaching the maximum etch rates of SiN_x and SiO_y at ~ 900 and ~ 8.1 Å/min, respectively. Note that, the etch selectivity of SiN_x over SiO_y didn't vary significantly (~ 120) over rf powers of 100 $\sim$ 400 W. In fact, as shown in figure 2 (b), the SiN_x and SiO_y could be also etched just by flowing ClF_3 gas only without dissociating ClF_3 by rf plasmas and the increase of substrate temperature increased the etch rates. However, the overall SiN_x etch rates by ClF_3 gas flow only were much lower compared to etching with ClF_3 remote plasmas, and which demonstrates that ClF_3 remote plasma etching is much more effective method for SiN_x etching compared with that by thermal etching without plasma. Meanwhile, even though etch rates of both materials were increased with increasing the substrate temperature, the etch selectivity of SiN_x over SiO_y was decreased. Therefore, as shown in figure 2c), when the etch rates of SiN_x and SiO_y were measured as a function of rf power at the substrate temperature of 100 °C, the similar trend was observed as those in figure 2a) while showing higher the SiN_x etch rates (above 2000 Å/min at 400 W of rf power) and lower etch selectivities (~ 60 at 400 W of rf power). Also, as shown in figure 2d), the further increase of substrate temperature to ~ 500 °C at a fixed rf power of 300 W showed the further decreases of etch selectivity below 40 while showing increased SiN_x etch rates over 6000 Å/min. Figure 3a) shows the selected etch rates of SiN_x and SiO_y for chemical etching with ClF_3 gas only at 100 °C, ClF_3 remote plasma etching (300 W) at RT, and ClF_3 remote plasma etching at 100 °C in figure 2 to illustrate the effect of plasma more clearly. As shown in figure 3a), for the chemical etching of SiN_x with ClF_3 , it clearly showed that the addition of remote plasma to thermal etching significantly increased the etch rate of SiN_x without decreasing the etch selectivity over SiO_y significantly due to the more reaction species on the surface by reactive radicals generated by the remote plasma. In addition to the role of the remote plasma, the role of process temperature on the etching of

SiN_x and SiO_y can be understood by plotting the etch rates of SiN_x and SiO_y logarithmically as a function of inverse temperature (1/T) for ClF₃ remote plasma etching as shown in figure 3b). For the chemically activated etching, the etch rates can be described as a following Arrhenius equation.

$$\ln k = - \frac{E_a}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right)$$

where k is a rate constant, R is the gas constant (8.31 J K⁻¹ mol⁻¹), and E_a is the activation energy. The calculated activation energies (E_a) of SiN_x and SiO_y were 3.09×10^{-20} and 5.08×10^{-20} J/mole, respectively. The higher activation energy of SiO_y means that the etch rate of SiO_y rises faster than that of SiN_x with the increase of temperature, and which leads to the decreases in etch selectivity of SiN_x over SiO_y even though the etch rates of both materials are increased exponentially with increasing substrate temperature. When the RMS surface roughness values of SiN_x and SiO_y were measured before and after the etching of ~ 400 nm and ~ 20 nm, respectively, with the etch methods in figure 3a), no significant differences in RMS surface roughness among the etch methods could be observed for both SiN_x and SiO_y (shown in figure S1, supplementary information).

To improve the etch selectivity of SiN_x over SiO_y, H₂ was added to ClF₃ in addition to Ar (Ar was also added to ClF₃/H₂ for plasma stability) and, the effect of H₂ addition to ClF₃ on the etch characteristics of SiN_x and SiO_y was investigated as a function of H₂ percentage in ClF₃/H₂ (ClF₃/H₂/Ar plasma) and the results are shown in figure 4a). To increase the H₂ percentage in ClF₃/H₂, H₂ flow rate was increased while keeping the substrate temperature at 25 °C, operating pressure at 200 mTorr, the ClF₃ flow rate at 200 sccm, Ar flow rate at 200 sccm, and the rf power at 300 W. As shown in figure 4a), the etch rates of both SiN_x and SiO_y were decreased with the increase of H₂ percentage, however, the etch selectivity of SiN_x over SiO_y was increased with the increase of H₂ percentage in ClF₃/H₂. To study the mechanism on the etching of SiN_x and the etch selectivity over SiO_y, the dissociated species in the plasmas and the byproducts at the pumping site were observed using OES and FTIR, respectively. Figure 4b) and c) shows optical emission spectra and the relative emission peak intensities of Cl, F, and H normalized by the intensity of Ar as a function of H₂ percentage in ClF₃/H₂, respectively. In figure 4b), the optical emission peak intensities related to Cl, H, F, and Ar could be measured at 280, 656, 704, and 750 nm, respectively. In figure 4c), the optical emission intensities of Cl, F, and H were normalized by the optical emission intensity of Ar (750 nm) to minimize the effect of electron density on the estimation of radical density from the emission intensity. As shown in figure 4c), the increase of H₂ percentage did not change the intensity of Cl, however, it decreased F intensity while increasing H intensity. Figure 4d) shows the FTIR data of the byproduct gases such as SiF₄ and HF measured at the pumping site for different H₂ percentage in ClF₃/H₂. As the flow rate of H₂ is increased, the concentration of SiF₄ was decreased while increasing HF concentration due to the reaction of hydrogen (H) with fluorine (F) radical in the plasma.

Therefore, from the figure 4b), c), and d), it is found that the decrease of etch rates of both SiN_x and SiO_y are related to the scavenging F radicals in the plasma by the formation of HF[19], which has negligible effects on the etching of SiN_x unlike its aqueous (ionic) state[20, 21], in the plasma with increasing H₂ percentage.

The Si binding states and compositions of the surfaces of SiN_x and SiO_y during the ClF₃/H₂ plasma etching were analyzed using X-ray Photoelectron Spectroscopy (XPS) and the results are shown in figure 5a-c) and d-f) for SiN_x and SiO_y, respectively, and also in Table 1. SiN_x and SiO_y were etched at the substrate temperature of 25 °C, operating pressure at 200 mTorr, the ClF₃/H₂/Ar flow rates at 200/(0 and 40)/200 sccm, and the rf power at 300 W. After the etching with ClF₃ plasma, significant Si-F bonding (103.6 eV) was formed on the SiN_x surface, presumably due to the bonding of Si with F (figure 5b). The Si-F bonding ratio decreases with addition of H₂ (20%) because of the reduction of F in the plasma (figure 5c and Table 1). However, no chlorine or Si-Cl bonding (~ 103.3 eV) was observed on the surface of SiN_x even though there were enough Cl radicals in the ClF₃/H₂ plasma as confirmed through OES data in figure 4c), presumably, due to the immediate reaction of Si-Cl with F radicals. Meanwhile, as shown in figure 5e, f), there was no significant change in F concentration on the SiO_y surface during etching with ClF₃ and ClF₃/H₂ plasma. Also, no noticeable Si-F bonding formation on the SiO_y surface during the etching with ClF₃ and ClF₃/H₂ plasma was observed from the deconvolution of Si narrow scan data (Si 2p) indicating that most of F is adsorbed on the SiO_y surface after the etching. Furthermore, the amount of F on the SiO_y surface is much lower than that of SiN_x because Si-O bonding is less reactive with F radical compared with SiN_x. The parameters used for curve fitting of SiN_x is described in Table 1 and the normalized chi-square value for curve fitting was below 0.01. The compositional information of each element can be found in Table S1, supplementary information.

Table 1

Parameters related with the curve fitting of silicon nitride (SiN_x) thin films after exposure to the ClF₃ only and ClF₃ & H₂ (20%) plasma

Sample	Binding state	B.E. (eV)	FWHM (eV)	% Area	Gaussian %
SiN _x (ClF ₃ only)	Si 2p	99.7	1.3	1.6	88.8
	Si-N	101.7	1.7	84.5	87.5
	Si-F	103.6	1.55 (±0.05)	13.9	85.7
SiNx [ClF3 & H2 (20 %)]	Si 2p	99.7	1.3	1.6	88.8
	Si-N	101.7	1.7	88	87.5
	Si-F	103.6	1.55 (±0.05)	10.4	85.7

The etching of SiN_x and SiO_y can be explained through the bonding energies of silicon (Si) compounds. Figure 6 shows the etch mechanism of SiN_x and SiO_y under Cl, F radicals. As the bonding energy of Si-F (565 KJ/mol) is higher than those of Si-N (355 KJ/mol) and Si-O (452 KJ/mol)[22], the SiN_x and SiO_y can be etched spontaneously under sufficient F radicals in the plasma although the etching is much active for SiN_x than SiO_y . However, the bonding energy of Si-Cl (381 KJ/mol) is slightly higher than that of Si-N but lower than that of Si-O, and which means the Cl radical can react only with SiN_x and forms Si-Cl bonding. Once the Si-N changes to Si-Cl, Si-Cl can be more easily converted to Si-F by F radicals in the plasma (due to the quick conversion of Si-Cl to Si-F as shown in figure 5, no chlorine could be observed on the surfaces of SiN_x and SiO_y during the etching with ClF_3/H_2), then Si-F on SiN_x is removed as a volatile SiF_4 compound. Meanwhile, the addition of H_2 in the ClF_3 plasma reduces the density of F radicals by forming HF in the plasma causing the decreases of Si-F formation on the surfaces of SiN_x and SiO_y , and which results in the decrease of etch rates of SiN_x and SiO_y . However, because the concentration of chlorine in the plasma is not significantly affected by the addition of H_2 as confirmed through OES data in figure 4c), the etching of SiN_x is decreased more slowly compared to that of SiO_y with increasing H_2 percentage through the conversion of Si-Cl on the surface of SiN_x to Si-F, and which appears to increase the etch selectivity of SiN_x over SiO_y .

Using the etch conditions of ClF_3 and ClF_3/H_2 (20%), stacked layers of $\text{SiN}_x/\text{SiO}_y$ were etched and the results are shown in figure 7. Figure 7a) is the reference stack of $\text{SiN}_x/\text{SiO}_y$ before the etching. Figure 7b) and c) are the stacked layer of $\text{SiN}_x/\text{SiO}_y$ after the etching using ClF_3 and ClF_3/H_2 (20%) plasmas for 5 min and 10 min, respectively. As shown in figure 7b) and c), highly selective etching of SiN_x over SiO_y could be observed for both ClF_3 and ClF_3/H_2 (20%) by showing no noticeable differences in SiO_y thickness along the etch depth. Therefore, it appears that the etch selectivity for the real $\text{SiN}_x/\text{SiO}_y$ could be higher than that measured with blank wafers. The etch depth with increasing the etch time was also measured and the results are shown in d) for both ClF_3 and ClF_3/H_2 (20%). The etch depth with etch time was linear for both conditions, therefore, no aspect ratio dependent etching was observed. (The process time-dependent etch profiles of $\text{SiN}_x/\text{SiO}_y$ stacks are shown in figure S2 and S3, supplementary information).

4. Conclusion

The isotropic and selective etching of SiN_x over SiO_y was studied using ClF_3/H_2 remote plasma with an ICP source. The SiN_x etching using plasma assisted thermal processes showed the highest etch rate as well as the smoothest surface morphology compared with that etched only with thermal etching or plasma etching. During the plasma etching, the etch rate of SiN_x was increased with increasing process temperature but the selectivity over SiO_y was found to be decreased due to the higher activation energy of SiO_y compared with that of SiN_x with ClF_3 plasma. The addition of H_2 (20%) to the ClF_3 plasma improved the etch selectivity of SiN_x over SiO_y from 130 to 200 even though the etch rate of SiN_x was decreased

from ~830 to ~230 Å/min. We believe the fast and ultra-high selective SiN_x etching technology can be applied not only to next generation 3D NAND process but also to various semiconductor processes where precise etching of SiN_x is required.

Declarations

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Conflicts of interest

There are no conflicts to declare.

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Figures

Figure 1

Schematic drawing of a remote-type inductively coupled plasma (ICP) etcher. At the center of the chamber, double grids having multiple holes are installed to prevent an ion bombardment and to deliver radicals only to the substrate. During the process, the substrate temperature was controlled (RT ~ 500 °C) by a silicon carbide (SiC) heater located below the substrate.

Figure 2

Etch characteristics of SiN_x and SiO_y a) as a function of rf power for ClF₃ remote plasma at RT, b) as a function of substrate temperature for chemical etching with ClF₃ gas flow only, c) as a function of rf power for ClF₃ remote plasma at the substrate temperature of 100 °C, and d) as a function of substrate temperature for ClF₃ remote plasma at 300 W of rf power. 200 sccm Ar (200 sccm) was added to ClF₃ for plasma stability.

Figure 3

a) Selected etch rates of SiN_x and SiO_y for chemical etching with ClF₃ gas only at 100 °C, ClF₃ remote plasma etching (300 W) at RT, and ClF₃ remote plasma etching at 100 °C in figure 2. b) logarithm etch rates versus 1/T for ClF₃ remote plasma etching of SiN_x and SiO_y in figure 2 for the extraction of the activation energies.

Figure 4

a) Etch characteristics of SiN_x and SiO_y with ClF_3/H_2 plasma as a function of H_2 percentage in ClF_3/H_2 . b) OES data of $\text{ClF}_3/\text{H}_2/\text{Ar}$ plasma with different H_2 percentage in ClF_3/H_2 . c) optical emission intensities of Cl, F, and H normalized by the intensity of Ar (750 nm) in b) plotted as a function of H_2 percentage. d) FTIR data of ClF_3/H_2 plasma during SiN_x etching. For ClF_3/H_2 remote plasmas, 200 sccm of Ar was added for the plasma stability.

Figure 5

XPS narrow scan (Si 2p) data of SiN_x (a-c) and SiO_y (d-f) after etching with remote ClF_3/H_2 plasma.

Figure 6

Schematic of chemical reaction of ClF_3/H_2 remote plasma on etching of SiN_x and SiO_y . Possible reaction paths are illustrated.

Figure 7

Etch characteristics of ClF₃ only and ClF₃/H₂ (20 %) plasma in stacked SiN_x/SiO_y. a) SEM images of reference stacked SiN_x/SiO_y. Etch profile of stacked SiN_x/SiO_y after the etching with b) ClF₃ plasma and c) ClF₃/H₂ (20 %) for 5 min and 10 min, respectively. d) Etch depth of SiN_x in the stacked SiN_x/SiO_y with etch time for ClF₃ and ClF₃/H₂ (20%) plasmas.

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