Etching organic low dielectric film in ultrahigh frequency plasma using N_2/H_2 and N_2/NH_3 gases

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The behavior of species in the gas phase and their effects on the etching characteristics of organic film with the lower dielectric constant (low-*k*) were investigated in 500 MHz ultrahigh frequency (UHF) plasma using N_2/H_2 and N_2/NH_3 gases. The absolute H and N radical densities and NH_3 molecule density in etching plasmas were evaluated by the vacuum ultraviolet absorption spectroscopy and the infrared diode laser absorption spectroscopy technique, respectively. The effects of frequency that excited the plasma in the production of H and N radicals were clarified by comparing the behavior of radicals in the UHF plasma with that in the inductively coupled plasma (ICP) exciting at the frequency of 13.56 MHz reported in the previous study. H radicals were produced more efficiently from NH_3 gas and N radicals more efficiently from N_2 gas in the UHF plasma than in the ICP. H radicals were generated not only through the dissociation of the H_2 molecule but also from NH_3 molecules produced in the N_2/H_2 plasma. On the other hand, the N radical density was lower by one order of magnitude than the H radical density, and was seldom produced from the NH_3 molecule. It was found that the etch rate and etched profile could be controlled with an internal plasma parameter of the ratio of the H and N radical densities under the constant electron density in the plasma gas chemistry of both N_2/H_2 and N_2/NH_3. The optimal conditions for the anisotropic profile and high etch rate were well determined by the ratio of the H and N radical densities and the substrate temperature. The results of this study are very useful not only for understanding the fundamental process of organic low-*k* film etching but also for precisely controlling the etching process. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1588351]

I. INTRODUCTION

As the packing density of the ultralarge scale integrated circuits increases, the linewidth and space between metal interconnections become smaller. The resistance capacitance (RC) delay becomes a major limitation in device performance due to the increase of the parasitic resistance and capacitance interconnections. To reduce the RC delay, the resistance of wiring metals and the parasitic capacitance of interlayer dielectrics must be reduced. Therefore, insulating materials with a lower dielectric constant (low-*k*) are available and are still being developed.[1–6] Various kinds of organic, inorganic, hybrid material, and these porous materials, have been proposed, and can be fabricated by either chemical vapor deposition or spin-on techniques. The nonfluorinated organic low-*k* material is one of the most promising materials because it has glass-transition temperatures higher than 450 °C, no outgassing below 400 °C, good adhesion, and gap-filling ability.[7] The etching process for organic low-*k* films can be performed without employing perfluorocarbon gases such as C_4F_8 of high global warming potential values. It has been reported that the high-density plasma employing N_2/H_2 and NH_3 gases are used for etching organic low-*k* films without degrading the film quality and the etched profile.[8–10] However, the kinetics of radicals in plasma and their influence on the etching performance have not been sufficiently clarified.

So far, etching characteristics have been investigated by the conventional external parameters such as gas pressure, power, and so on. However, the results obtained using the conventional parameters do not result in common fundamental information. Therefore, the etching of organic low-*k* films should be performed using the internal plasma parameters such as electron density and temperature, radical and ion density without using external parameter. Moreover, to obtain high etching performance, information on etching properties defined by the internal plasma parameters is very useful and applicable to various etching apparatus.

In our previous study, this organic low-*k* film, FLARE™, was etched in inductively coupled plasmas (ICPs) operating at 13.56 MHz employing N_2/H_2 and N_2/NH_3 gases. The etching plasmas were evaluated by quadruple mass spectroscopy and vacuum ultraviolet absorption spectroscopy (VUVAS) employing microplasma as a light source. From these results, the behavior of the etch rate of an organic low-*k* film corresponded well to that of H radical densities in the N_2/H_2 and N_2/NH_3 plasma. H radicals were found to be an important species for organic low-*k* film etching, while N radicals could not etch the film without ion bombardments. On the other hand, N radicals were found to be effective in the formation of a protective layer on the sidewall of the etched pattern against etching by H radicals.[11]

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The physical and chemical aspects in plasma are greatly influenced by the electron energy distribution function (EEDF), which is strongly related with the frequency of the plasma generation. Therefore, the selection of frequency is the most important factor in obtaining high etching performance and it is very interesting from a scientific viewpoint to investigate the kinetics of radicals produced at varying frequencies that excite plasma and thereby to relate etching performance with the behavior of radicals. This study investigates the etching of organic low-k films in an ultrahigh frequency [(UHF): 500 MHz] plasma employing N_2/H_2 and N_2/NH_3 gases. UHF plasma, where no magnetic field is required to maintain high-density plasma, has great potential in achieving high density with low electron temperature all over a large area of 30 cm diameter at low pressure. Furthermore, UHF plasma has been reported to have a large amount of electrons distributed in the range from a few eV to 10 eV and, moreover, whose electron population is relatively large at high-energy tails of above 10 eV.[12] To investigate the behavior of the species, the VUVAS technique and the infrared diode laser absorption spectroscopy (IR-LAS) technique were applied to measure the absolute densities of H (Refs. 13 and 14) and N (Ref. 15) radicals and NH_3 molecular density in etching plasma, respectively. The correlation between the behavior of the N and H radicals, and the NH_3 molecule in UHF plasma and the etching characteristics of organic low-k film were investigated, and compared to the previous results of ICP operating at 13.56 MHz. The characteristics of the etch rate and the etched profile are discussed using an internal plasma parameter of the ratio of the H and N radical densities.

II. EXPERIMENT

Figure 1 shows a schematic diagram of an UHF plasma etcher of 8-in.-wafer production level (ANELVA: I-4100) equipped with the IR-LAS system and the VUVAS system employing the microdischarge hollow-cathode lamp (MHCL) used in this study. The UHF plasma chamber was 40 cm in diameter and 30 cm in height. A six spoke antenna with a diameter of 30 cm was set on a quartz window at the top of chamber. Plasma power operating at 500 MHz was applied to the antenna. The gap between the wafer and the quartz top plate was fixed at 100 mm. The gas inlet pipe was ring shaped with eight holes, and was installed in the upper side of the chamber. A wafer was placed on the stage with an electrostatic chuck system. The substrate temperature was controlled in the range of 0–80 °C with a cooling solvent. The measurement of the plasma parameter and radical species was performed at 40 mm below the quartz top plate. The absolute H and N radical densities were measured using the VUVAS technique. The vacuum ultraviolet (VUV) light from the MHCL was made parallel and introduced into the chamber through an MgF_2 lens. The detailed structure of MHCL is described in Ref. 13. The VUV light was passed once through the chamber at 60 mm above the substrate, and was focused on the slit of a VUV monochromator (Acton Research Corp., ARC VM-520) by the MgF_2 lens and detected by a photomultiplier tube. The signal was averaged by a digital oscilloscope and recorded by a personal computer.

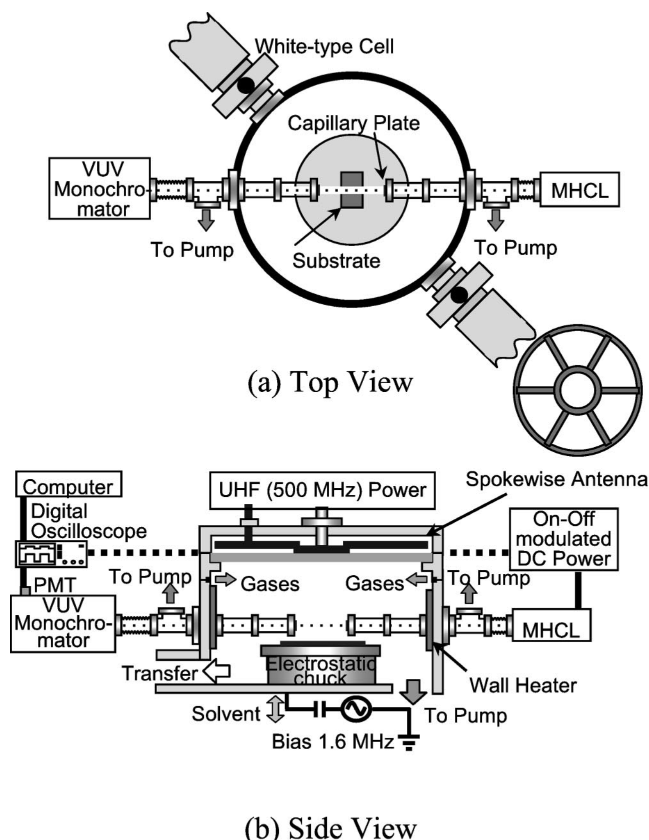

FIG. 1. Schematic diagram of UHF plasma etcher 8-in.-wafer production level (ANELVA: I-4100) equipped with the VUVAS system employing MHCL.

The transition lines used for absorption measurements were a Lyman α at 121.6 nm for the H radical, and $^4P_{5/2}-^4S^0_{3/2}$, $^4P_{3/2}-^4S^0_{3/2}$, and $^4P_{1/2}-^4S^0_{3/2}$ at 120.0 nm for the N radical, respectively. In order to calculate the absolute density in the VUVAS measurement with high accuracy, it is necessary to estimate background absorption due to the other molecules and radicals produced in the plasma.[14–16] The background absorption was evaluated by the broad peak of an Ar excimer at 122.5 nm. The absolute densities of the H and N radicals were estimated by subtracting the background absorption from the total absorption intensity as described in Ref. 16. The NH_3 molecular density was measured using the IR-LAS technique. The absorption line of the NH_3 molecule used in this study was $aR(6,1)$ at 1075.8288 cm^{-1}.

The etching conditions were maintained at a total pressure of 2 Pa and a total flow rate of 100 sccm. The self-bias voltage (V_{dc}) was fixed at -500 V by adjusting the rf bias power (1.6 MHz). The cross section of etching profile was examined by using a field emission electron microscope (HITACHI, S-4300).

III. RESULTS AND DISCUSSION

A. Characteristics of ultrahigh frequency N_2/H_2 and N_2/NH_3 plasma

Figure 2 shows electron densities measured by a 35 GHz microwave interferometer as a function of the element ratio

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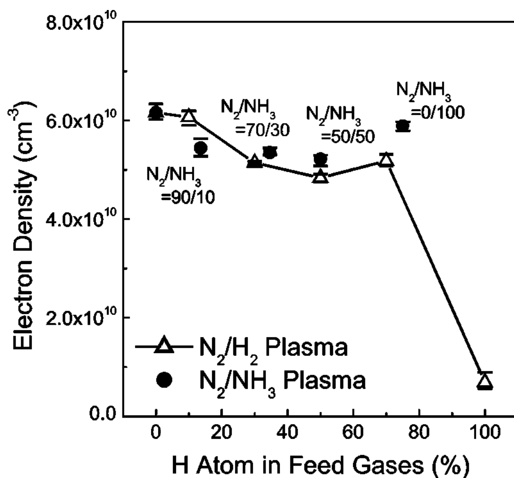

FIG. 2. Electron densities measured by a 35 GHz microwave interferometer as a function of the H atom in feed gases, in UHF plasma employing N_2/H_2 and N_2/NH_3 gases.

of the H atom to the sum of the H and N atoms involved in the chemical structure of N_2/H_2 and N_2/NH_3 feed gases. For example, the H atom in feed gases is 75% when under $N_2/NH_3=0/100$ sccm. The plots in Fig. 2 indicate the averages of five measurements, and the error bars correspond to the maximum and minimum values. In the case of N_2/H_2 plasma, the electron density in the pure N_2 plasma, corresponding to the H atom in the feed gases=0%, is the highest. As the H atom in the feed gases increased, the electron densities were almost constant. The electron density drastically decreased in the H_2 plasma. This is due to the small total ionization cross section of H_2, which is about 2.5 times lower than that of N_2. On the other hand, the electron densities were almost constant in the N_2/NH_3 plasma because the total ionization cross section of N_2 and NH_3 are almost the same.[17]

Figure 3 shows the etching characteristics of FLARE™ as a function of the H atom involved in feed gases. The etching was carried out on the blanket film of FLARE™. The substrate temperature was fixed at 20 °C. In Fig. 3, the open triangles (△) represent the etch rate using the N_2/H_2 plasma,

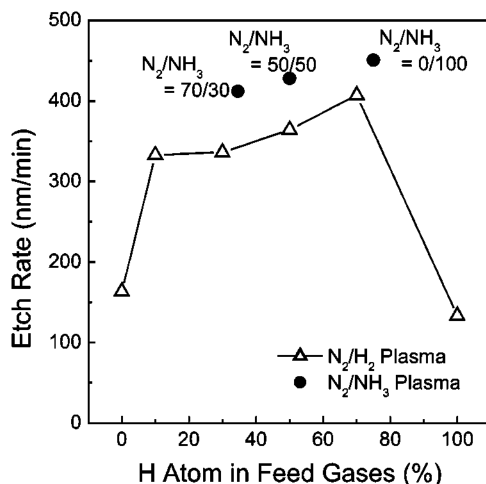

FIG. 3. Etch rate of FLARE™ as a function of the H atom in feed gases.

and the filled circles (●) represent the etch rate using the N_2/NH_3 plasma. In the N_2/H_2 plasma, as the H atom in the feed gases increased, the etch rate increased, reaching a maximum of 400 nm/min in H atom in the feed gases=0.7 ($N_2/H_2=30/70$ sccm), and then decreased. In the N_2/NH_3 plasma, as the H atom in the feed gases increased, the etch rate increased, and reached a maximum of 450 nm/min at H atom in the feed gases=0.75 ($N_2/NH_3=0/100$ sccm). The etch rate of the N_2/NH_3 plasma was higher by about 10% than that of the N_2/H_2 plasma in spite of constant electron density. In our previous study using ICP, the etch rate depended on the ratio of the H atom in the feed gases, and was independent of the kind of gases under these etching conditions.[11] In the case of the UHF plasma, the etch rate in the N_2/NH_3 plasma showed behavior similar to that in the N_2/H_2 plasma in its variation in H atom ratio in the N_2/H_2 and N_2/NH_3 gases. However, the etch rate in the N_2/H_2 and N_2/NH_3 plasma was not determined solely by the H atom in the feed gases as shown in Fig. 3.

Figures 4(a) and 4(b) show the N and H radical densities measured using the VUVAS technique as a function of the H atom in the feed gases in the UHF plasma [Fig. 4(a)] and in ICP [Fig. 4(b)] reported in Ref. 11. In Figs. 4(a) and 4(b), the open symbols (○, △) represent the H and N radical densities in the N_2/H_2 plasma, and the filled symbols (●, ▲) represent the H and N radical densities in the N_2/NH_3 plasma, respectively. The electron densities in the UHF N_2/H_2 and N_2/NH_3 plasmas were the same as those in the previous ICP experiment to adjust the UHF power. With the UHF N_2/H_2 plasma as shown in Fig. 4(a), as the H atom in the feed gases increased, the absolute H radical densities increased, reaching a maximum of 1.3×10^{13} cm^{-3}, then decreased at a pure H_2 plasma. This decrease is due to the electron density that was lower by one order of magnitude at the pure H_2 plasma as shown in Fig. 2. Compared with the results in ICP as shown in Fig. 4(b), the H radical densities in the UHF N_2/H_2 plasma were almost the same as those of the ICP N_2/H_2. On the other hand, the N radical densities increased gradually with a decrease in the H atom in the feed gases. It is noteworthy that the N radical densities in UHF N_2/H_2 plasma were twice as high as those of the ICP N_2/H_2. In the case of the UHF N_2/NH_3 plasma, as shown in Fig. 4(a), as the H atom in the feed gases increased, the absolute H radical densities increased, reaching a maximum of 5.0×10^{13} cm^{-3} at the pure NH_3 plasma. Unlike ICP, the H radical densities in the UHF N_2/H_2 and N_2/NH_3 plasma were not determined by the H atom in the feed gases as shown in Figs. 4(a) and 4(b). It is noted again that the H radical densities in UHF N_2/NH_3 plasma were much higher than those of the ICP. This behavior of the H and N radicals in the UHF plasma is explained by the difference in EEDFs between that in the ICP and the UHF plasmas. The dissociation cross-sections of the N_2 gas by electron collision is quite a lot larger than that of H_2, though the dissociation of the N_2 gas needs higher electron energies above 10 eV.[18,19] Moreover, the dissociation cross-section of the NH_3 molecule at the lower electron energy is thought to be larger than those of the H_2 and N_2 gases. On the other hand, it has been reported that the EEDFs in ICP and UHF plasmas are very different.[12] In the case of

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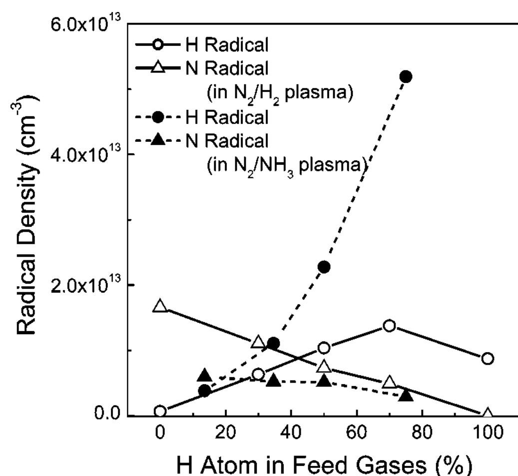
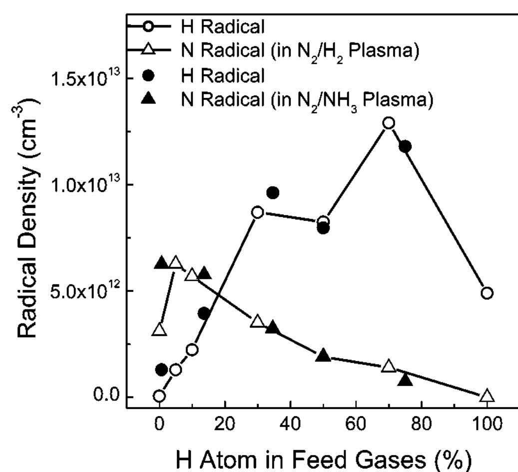


(a) H and N radical densities in UHF plasma



(b) H and N radical densities in ICP[11]

FIG. 4. N and H radical densities measured using the VUVAS technique as a function of the H atom in feed gases in UHF plasma (a) and in ICP (reported in Ref. 11) (b).

UHF plasma, the non-Maxwellian EEDF can be formed. It has been reported that a large amount of electrons are distributed in the range from a few eV to 10 eV and, moreover, the electron population is relatively large at high-energy tails of above 10 eV.[12] Therefore, the distribution of electrons in high-energy tails of above 10 eV in UHF plasma would result in the efficient generation of the N radical from N_2 gas. In addition, the efficient generation of the H radical from NH_3 is attributed to the majority of relatively low-energy electrons together with higher-energy electrons, which is due to the specific EEDF of UHF plasma.

Figure 5 shows NH_3 molecular densities in an UHF plasma as a function of the H atom in feed gases employing the IR-LAS technique. In the N_2/H_2 plasma, as the H atom in the feed gases increased, the absolute NH_3 molecular densities increased, reaching a maximum at the H atom in the feed gases of 70%, that is $N_2/H_2=30/70$ sccm, then decreased. In the N_2/NH_3 plasma, as the H atom in the feed gases increased, the NH_3 molecular densities increased,

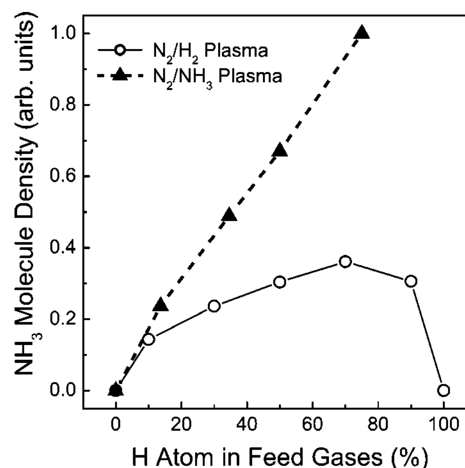


FIG. 5. NH_3 molecular densities measured using IR-LAS technique as a function of the H atom in feed gases.

reaching a maximum at the H atom in the feed gases of 75%, that is, pure NH_3 plasma. The behavior of NH_3 molecular density was similar to that of the H radical densities as shown in Fig. 4(a). The NH_3 molecular densities under these conditions were estimated on the order of 10^{13} cm^{-3} using the line strength calculated from the NH_3 gas at 2 Pa when the gas temperature of the NH_3 molecule is assumed to be 300 K. It was found that a large amount of NH_3 molecules were synthesized in the N_2/H_2 plasma. From these results, in the N_2/H_2 plasma, the H radical would be generated not only from the H_2 molecule but also from the NH_3 molecule produced in plasma by electron impact dissociation. On the other hand, less N radicals would be produced from the NH_3 molecule with electron impact dissociation in the present conditions.

B. Evaluation of etch rate and etched profile using an internal plasma parameter

The etch rate of FLARE™ employing UHF plasma etching, as shown in Fig. 3, was not dependent on the H atom in the feed gases. Therefore, the etch rate was replotted as a function of the internal plasma parameter that is proposed in this study. Figure 6 shows the etch rate as a function of the ratio of H and N radical density, $H/(H+N)$, measured by that VUVAS technique in the UHF plasma. It is considered that the ratio of H and N radical density is the most important value related to the coverage of the active site in the etched subsurface. In Fig. 6, the open triangles (△) represent the etch rate in the N_2/H_2 plasma, and the filled circles (●) represent the etch rate in the N_2/NH_3 plasma. In both the N_2/H_2 and N_2/NH_3 plasma, as the ratio of H and N radical densities in the plasma increased, the etch rate linearly increased under the constant electron densities as shown in Fig. 2. The large deviation in etch rate from the line at pure H_2 plasma results from the electron density being lower by one order magnitude at the H_2 plasma, as shown in Fig. 2, because the electron density is nearly equal to the ion density that causes the etching subsurface reaction. Furthermore, in the case of ICP etching, a linear relation between etch rate and the ratio of H and N radical densities measured by the

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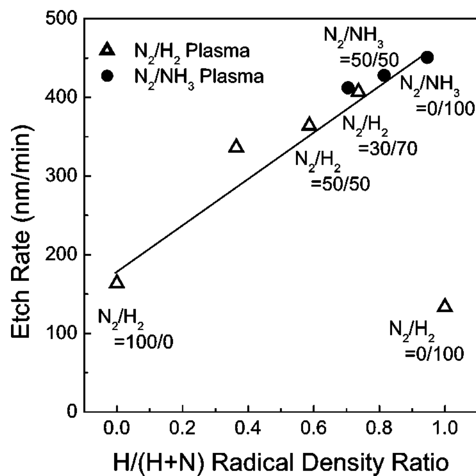

FIG. 6. Etch rate of FLARE™ as a function of the H and N radical density ratio.

VUVAS technique was observed. As a result, it was found that there was an obvious quantitative relation between the etch rate and the ratio of H and N radical density even when the different mixtures of N_2/H_2 and N_2/NH_3 gases were employed. Consequently, the etch rate of an organic low- k film can be determined by the ratio of H and N radical densities under constant electron density, independent of the kind of gas. This indicates that the etch rate is determined at a balance of reaction of enhancement of etching by H radicals and suppression of chemical etching by N radicals without ion bombardment as described in previous study.[11]

The etched profile in UHF plasma was investigated at different substrate temperatures. The samples were etched for 1 min in N_2/H_2 and N_2/NH_3 plasmas at a total pressure of 2 Pa, a total flow rate of 100 sccm, and a self-bias voltage of -500 V. The patterned sample, which has a pattern size of a 0.3 μm line and space structure, was etched under the same conditions as those used for the etch blanket FLARE™. The sample used in this experiment consists of a photoresist/SiO_2/FLARE™/SiO_2 stacked structure. The thickness of the hard mask, that is SiO_2, was 200 nm and the thickness of FLARE™ was 500 nm.

Figure 7 shows the amount of bowing at the sidewall of the line pattern as a function of the ratio of H and N radical densities in N_2/H_2 and N_2/NH_3 plasmas measured by the VUVAS technique. The amount of bowing is defined as the difference between the width at the bowing position or the center point of the etched depth "B" and the width at the line at the top "A" as shown in Fig. 7. At the substrate temperature of 0 °C, the tapered profiles were observed at ratios of H and N radical densities below 0.6 and then the anisotropic profiles were obtained at ratios of H and N radical densities above 0.6. When the substrate temperature was 20 °C, the amount of bowing was constant and negligibly small for the variation in the ratio of H and N radical densities. Anisotoropic profiles were obtained over the wide range from the ratio of H and N radical densities =0.5 to 0.8. When the substrate temperature was above 20 °C, as the H radical density ratio increased, the amount of bowing was almost constant up to a ratio of H and N radical densities of 0.6, then

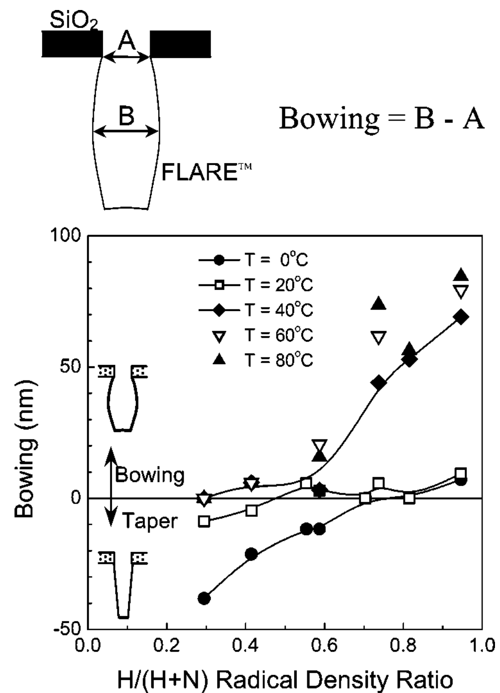

FIG. 7. The amount of bowing at the sidewall of the line pattern as a function of the H radical density ratio measured by the VUVAS technique in N_2/H_2 and N_2/NH_3 plasmas.

the amount of bowing increased linearly. From these results, the etch rate and etched profile characteristics could be controlled by the ratio of H and N radical densities, because they are decided dominant by the balance of chemical etching by the H radical and the protection layer formation by the N radical together with a byproduct, which is strongly dependent on substrate temperature. Moreover, it was found the process window could be extended by controlling substrate temperature.

By optimizing the conditions of the internal plasma parameter where the ratio of H and N radical densities = 0.8 (N_2/NH_3 = 50/50 sccm) in the plasma, and substrate temperature was 20 °C, the anisotropic profile with an etch rate of about 400 nm/min was obtained as shown in Fig. 8. The

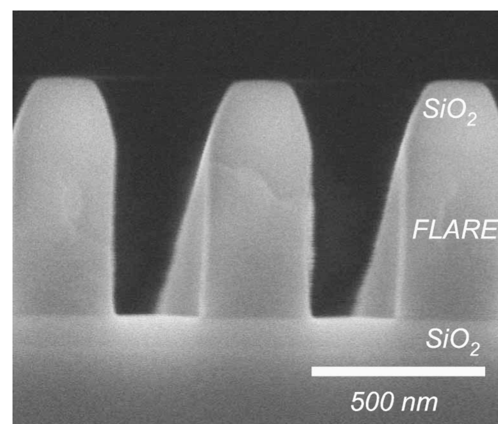

FIG. 8. SEM image of etched profile employing N_2/NH_3 plasma at H radical density ratio =0.8 (N_2/NH_3 = 50/50 sccm), and substrate temperature of 20 °C.

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scientific information resulting from the internal plasma parameter of the radical density ratio obtained in this study is very useful for developing the etching of organic films.

IV. CONCLUSIONS

Organic low-*k* film etching was carried out on 500 MHz UHF plasma employing N_2/H_2 and N_2/NH_3 gases. The correlation between the etching characteristics of organic low-*k* film and the kinetics of N and H radicals, and the NH_3 molecule in an UHF plasma were investigated using the VUVAS and IR-LAS techniques, respectively. It was found that the N and H radicals were efficiently generated from the N_2 and NH_3 gases, respectively, due to the specific EEDF of the UHF plasma. The behavior of the NH_3 molecular density corresponded to that of H radical density in N_2/H_2 and N_2/NH_3 plasmas. Therefore, in the N_2/H_2 plasma, the H radical would be generated not only from the H_2 molecule but also from the NH_3 molecule produced in plasma by electron impact dissociation. On the other hand, less N radicals would be produced from the NH_3 molecule with electron impact dissociation. Furthermore, the organic low-*k* film was etched by controlling H and N radical densities in the plasma under constant electron density, independent of feed gases. The etch rate and etched profiles could be controlled precisely by selecting the ratio of H and N radical densities and substrate temperature. By optimizing the condition of the internal plasma parameter of the radical density ratio $H/H+N=0.8$ and with a substrate temperature of 20 °C, the anisotropic profile with a high etch rate of about 400 nm/min was obtained. The ratio of H and N radical densities proposed as an internal plasma parameter was found to be the most important factor in precisely controlling the etching processes of organic films.

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