Surface reaction of CF_2 radicals for fluorocarbon film formation in SiO_2/Si selective etching process

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The surface reaction of CF_2 radicals on Si and fluorocarbon films was investigated in electron cyclotron resonance (ECR) Ar and H_2/Ar downstream plasmas employing CF_2 radical injection technique. The effects of Ar^+ ions, Ar* metastable species and radiation from plasmas on the fluorocarbon film formation were evaluated in ECR Ar downstream plasma with CF_2 radical injection. As a result, CF_2 radicals with assistance of Ar^+ ion bombardment were found to play an important role in the fluorocarbon film formation. The adsorptive reactions of CF_2 radicals on the fluorocarbon film surface with and without Ar and H_2/Ar plasma exposures were successfully investigated by *in situ* Fourier transform infrared reflection absorption spectroscopy and *in situ* x-ray photoelectron spectroscopy. It was found that the formation of fluorocarbon film in the plasma proceeded through the adsorptive reaction of CF_2 radicals at a high probability on the active sites formed by the bombardment of Ar^+ ions on the fluorocarbon film surface. © *1998 American Vacuum Society.* [S0734-2101(98)02801-2]

I. INTRODUCTION

In SiO_2/Si selective etching processes employing fluorocarbon plasmas, the fluorocarbon film formed on the Si strongly influences the etching selectivity. The importance of fluorocarbon film formation in the SiO_2/Si selective etching process has been extensively studied using several etching tools such as electron cyclotron resonance (ECR) plasma reactor employing CF_4 and CHF_3,[1] inductively coupled plasma (ICP) reactor employing C_4F_8/H_2 (Ref. 2) and CF_4/C_2H_4 (Ref. 3) by many groups. In order to realize a highly selective etching, it is necessary to clarify the formation mechanism of fluorocarbon film. So far, several researchers have studied the surface reactions of individual fluorocarbon radicals on Si and SiO_2 exposed to irradiation of laser or ion beam instead of the plasmas.[4–8] However, the experimental conditions were quite different from those in the plasma where the surfaces of substrates were exposed simultaneously to ions, electrons, and photons. Therefore, for the clarification of formation mechanism of fluorocarbon film, it is important to understand the surface reactions of the individual radicals under conditions of plasma processing.

It has been generally reported that CF_2 radicals were playing an important role for the fluorocarbon film formation.[1,9–11] However, the lifetime of CF_2 radical was relatively long and the measured decay lifetime of the CF_2 radical was about 500 ms in the parallel plate rf discharge plasma employing CHF_3 and CF_4/H_2, and in the ECR downstream plasma employing CHF_3.[12–14] Moreover, the self-sticking coefficient for CF_2 radicals was only on the order of 10^{-4}–10^{-3}.[6] On the other hand, it has been reported that the self-sticking coefficient for CF radicals was as large as 0.1.[15] Given these findings, it appears unlikely that CF_2 radicals contribute predominantly to the fluorocarbon film formation in the plasma.

In our previous study, we developed the radical injection technique in order to clarify the formation mechanism of fluorocarbon film in the plasma, and have investigated the properties of fluorocarbon films formed through CF_2 radicals on Si in ECR Ar or H_2/Ar downstream plasma with CF_2 radical injection.[16,17] In both ECR Ar and H_2/Ar downstream plasmas, the fluorocarbon films were formed by injecting CF_2 radicals on the Si surfaces without applying bias voltage. The deposition rate of fluorocarbon films increased linearly with increasing the microwave power. Moreover, the fluorocarbon film was not easily formed by CF_2 radicals without the plasma exposure. In this case, the deposition rate was <0.1 nm/min. These results indicated that with the assistance of plasma exposures the CF_2 radicals formed the fluorocarbon film at a high rate. However, it has not been clarified what factors in the plasma exposure, namely, charged species, metastable atoms, or light radiation from plasma, dominantly contribute to the fluorocarbon film formation from CF_2 radicals. Oehrlein *et al.* reported that the deposition rate of fluorocarbon film on the substrate increased with increasing ion bombardment in the ECR CHF_3 plasma.[1] However, the surface reaction of CF_2 radicals was not investigated enough in their study.

In our study, in order to evaluate the effects of Ar^+ ions from plasma on the fluorocarbon film formation in ECR Ar plasma with CF_2 radical injection, the deposition rate of fluorocarbon film on a Si substrate was examined by controlling the flux of charged species incident on the substrate with the

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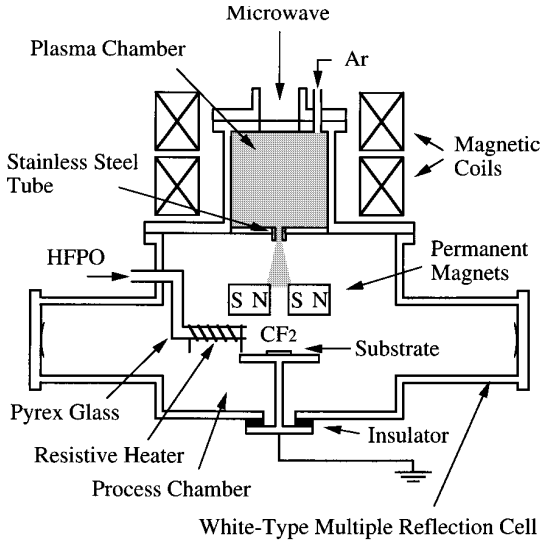

FIG. 1. ECR plasma process system equipped with CF_2 radical injection source.

use of permanent magnets. In addition, the effects of Ar atoms (Ar*) at metastable state and light radiation from plasma on the film formation were studied. Moreover, we studied the surface reaction of CF_2 radicals on the fluorocarbon film exposed to ECR Ar and H_2/Ar downstream plasmas employing CF_2 radical injection technique, *in situ* Fourier transform infrared reflection absorption spectroscopy (FTIR RAS), and *in situ* x-ray photoelectron spectroscopy (XPS).

II. EXPERIMENT

Figure 1 shows the ECR plasma process system equipped with CF_2 radical injection source. It consists of a stainless steel plasma chamber of 15 cm in diameter and 28 cm in height, and a stainless steel process chamber of 40 cm in diameter and 40 cm in height. The plasma was directed to the process chamber in the divergent magnet field produced by using two magnet coils. The ECR point was located at 10 cm below the top of the plasma chamber. The Ar gas was introduced into the top of the plasma chamber. White-type multiple reflection cell of 200 cm in length was installed at the process chamber to increase the absorption length of an IR laser beam used for the radical density measurement. The CF_x $(x=1-3)$ radical densities were measured using IR diode laser absorption spectroscopy (IRLAS). The laser beam was passed through the ECR downstream plasma 12 times at 2 cm above the substrate plate using White-type multiple reflection cell for the measurement of CF_2 radical density and 40 times for those of CF and CF_3 radical densities. The absorption lines used in this study were $R_1(7.5)$ and $R_2(7.5)$ lines for CF radicals,[18] ${}^{Q}R_4(26)$ line for CF_2 radicals,[19] ${}^{R}R_{18}(18)$ line for CF_3 radicals.[20] The measurement method of the CF_x $(x=1-3)$ radical densities using IRLAS has been described in detail in Ref. 14.

CF_2 radical source was set in the process chamber as shown in Fig. 1. The distance between CF_2 radical source and substrate was about 10 cm. CF_2 radicals were produced by the pyrolysis of hexafluoropropyleneoxide (HFPO, CF_3CFOCF_2) in a 1/4 in. Pyrex glass tube heated resistively, and were injected on to the substrate. HFPO was decomposed into the CF_2 radical and the stable CF_3CFO molecule at temperature above 600 K in Pyrex glass tube.[4,5] In order to prevent HFPO and decomposed species from pouring into the ECR region to be further dissociated, a stainless steel tube of 1.3 cm in diameter and 1 cm in length was set between the plasma chamber and the process chamber as shown in Fig. 1. Parent HFPO molecules, decomposed stable CF_3CFO molecules and CF_2 radicals were not dissociated by Ar plasma in the process chamber, judging from the facts that the CF and CF_3 radicals were not detected in the process chamber and the CF_2 radical density with the Ar plasma exposure was the same value as that without Ar plasma as a result of radical density measurement using IRLAS. Furthermore, HFPO and CF_3CFO are considered to be stable molecular species with low sticking coefficient, which would not adsorb on and react with the Si surface.[4,5] Therefore, it can be considered that the surface reaction of only CF_2 radicals was evaluated in this system. The fluorocarbon films were formed at HFPO flow rate of 12.5 sccm, Ar flow rate of 25 sccm, total pressure of 1.3 Pa, and microwave power of 800 W. The deposition period was 30 min. In this condition, CF_2 radical density at 2 cm above Si substrate was constant at 2×10^{13} cm^{-3} by heating the Pyrex tube up to 700 K. Although the substrate might be heated somewhat by heating the Pyrex tube, the substrate temperature was considered to be constant in all experiments. Therefore, an influence of the substrate temperature on the sticking probability of CF_2 radical was neglected in our study. The film thickness was measured by a tally step.

Moreover, for the evaluation of the effect of Ar^+ ions on the fluorocarbon film formation, a pair of permanent magnets were employed in the process chamber in order to bend Ar^+ ions away from Si substrate exposed to ECR Ar downstream plasma, as shown in Fig. 1. The magnets were located at 2 cm above the substrate. The dimensions of each magnet were $3\times3\times5$ cm^3. The magnetic flux density between two magnets was 0.3 T.

In order to investigate the influence of Ar* atoms in this system, the absolute density of Ar* $({}^3P_2)$ atoms was measured by using visible light absorption spectroscopy.[21] In this measurement, a pair of permanent magnets were removed, and Ar hollow cathode lamp was used as a light source. The Ar hollow cathode lamp was driven at Ar flow rate of 10 sccm, pressure of 266 Pa, dc voltage of 400 V, and dc of 0.08 A. In the case using the stainless steel tube of 1.3 cm in diameter between the plasma chamber and the process chamber, the density of Ar* $({}^3P_2)$ atoms was so low that the absorption of Ar* $({}^3P_2)$ atoms was not observed. Then, the diameter of the tube between the plasma and the process chambers was changed to 4 cm to increase the volume of the ECR Ar downstream plasma. ECR Ar plasma was generated at Ar flow rate of 12.5 sccm and pressure of 0.67 Pa. In this condition, the emitting region of Ar plasma generated in the process chamber was 20 cm in diameter near the substrate.

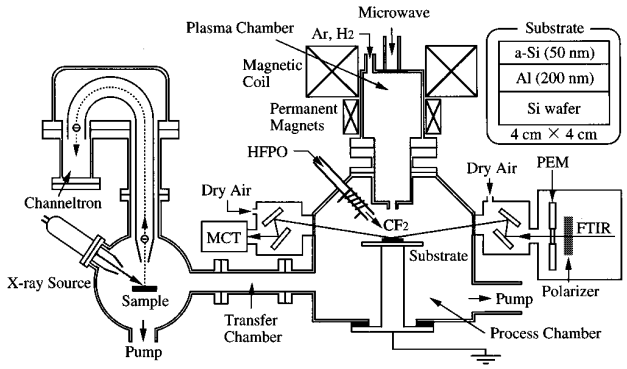

FIG. 2. ECR plasma processing system equipped with *in situ* FTIR RAS and XPS.

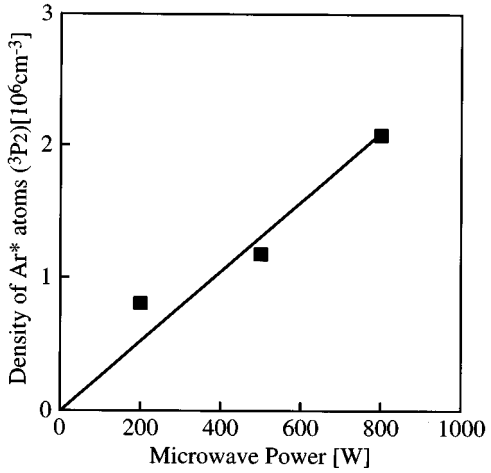

FIG. 3. Density of Ar* atoms ($^3P_2$) as a function of microwave power.

Therefore, the absorption length was 20 cm in the absorption measurement of Ar* ($^3P_2$) atoms.

For the evaluation of the surface reaction of CF_2 radicals in ECR Ar and H_2/Ar plasmas, another ECR plasma process system equipped with *in situ* FTIR RAS and *in situ* XPS was used, as shown in Fig. 2. It consists of a stainless steel plasma chamber of 15 cm in diameter and 16 cm in height, and a stainless steel process chamber of 40 cm in diameter and 40 cm in height. The magnetic field was produced by a magnetic coil and permanent magnets. The ECR point was located at about 10 cm below the top of the plasma chamber. The Ar or H_2/Ar mixture was introduced into the top of the plasma chamber. The substrate was set at 35 cm away from the ECR point. CF_2 radical source as described before was introduced in the process chamber as shown in Fig. 2. A stainless steel tube of 1 cm in diameter and 2 cm in length was set at 8 cm above the substrate.

In the second system, the process chamber was equipped with *in situ* FTIR RAS. IR beam was introduced through the polarizer, photo elastic modulator (PEM), and KBr window into the process chamber at an incident angle of 80°. The beam was reflected at the substrate and detected by HgCdTe (MCT) detector. Using this *in situ* FTIR RAS, the surface reaction of CF_2 radicals was observed in real time. The wavenumber resolution of FTIR RAS was 4 cm^{-1}. The substrate with buried metal structure as shown in Fig. 2 was employed in order to increase the sensitivity for FTIR RAS measurement. The triple-layered substrate consists of *a*-Si (top layer, 50 nm thickness), aluminum (Al) (200 nm thickness), and *n*-type Si(100) substrate. *a*-Si and Al films were deposited by the vacuum evaporation system with an electron beam (ULVAC EBV-6DH). The dimensions of the substrates were 4×4 cm^2.

Furthermore, XPS system was connected to the ECR chamber through a transfer chamber in a vacuum. In XPS system, Mg $K\alpha$ line was used as an x-ray source. The fluorocarbon films formed by ECR plasma exposure were transferred to XPS chamber without atmospheric exposure. Therefore, this *in situ* XPS analysis enabled us to investigate the chemical structure of fluorocarbon film surface immediately after a plasma exposure.

III. RESULTS AND DISCUSSION

A. Fluorocarbon film formation through CF_2 radicals with and without Ar^+ ions

At the magnetic flux density of 0 T, the deposition rate was 3.2 nm/min. On the other hand, at the magnetic flux density of 0.3 T, the thickness of the deposited film was <2 nm, which was, the value of measurement limit by a tally step. By applying the magnetic field at the magnetic flux density of 0.3 T, Ar^+ ions with kinetic energy <30 eV would be deflected. The current coming into substrate plate in the case using a pair of permanent magnets to deflect Ar^+ ions has been measured with a current meter in ECR SiH_4/H_2 plasma. As a result, the current density with permanent magnets was <3 nA/cm^2 while that without permanent magnets was 1 mA/cm^2, which indicated that most of Ar^+ ions in the ECR plasma were deflected by applying the magnetic field at the magnetic flux density of 0.3 T.[22] Therefore, it was deduced that CF_2 radicals formed the fluorocarbon film at a high rate on the Si surface only with assistance of ion bombardment from the plasma.

The contribution of photons and Ar* atoms in the plasma to fluorocarbon film formation is considered to be negligible in ECR Ar plasma with CF_2 radical injection. This conclusion was verified by the following experiment: In order to investigate the influence of Ar* atom in this system, the absolute density of Ar* ($^3P_2$) atoms was measured by using visible light absorption spectroscopy.[21] Figure 3 shows the density of Ar* ($^3P_2$) atoms as a function of microwave power without CF_2 radical injection. The Ar* ($^3P_2$) atom density increases with increasing microwave power, and is estimated to be of the order of 10^6 cm^{-3} at a microwave power of 800 W. The Ar* ($^3P_0$) atoms density was below the detection limit. Under the same condition, the electron density near the substrate in the process chamber was measured by a Langmuir probe. The electron density was estimated to be of the order of 10^8 cm^{-3}. Therefore, Ar^+ ion density was considered to be of the order of 10^8 cm^{-3}. Ar* density was lower by 2 orders of magnitude than Ar^+ ion

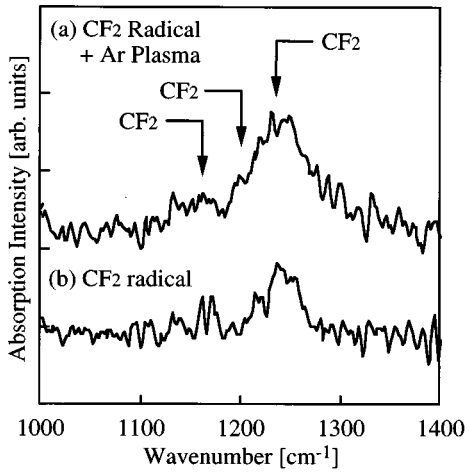

FIG. 4. FTIR spectra of (a) the fluorocarbon film formed on the *a*-Si surface in the ECR Ar downstream plasma with CF_2 radical injection and (b) the fluorocarbon film formed on the *a*-Si surface by CF_2 radical injection without the ECR Ar downstream plasma exposure.

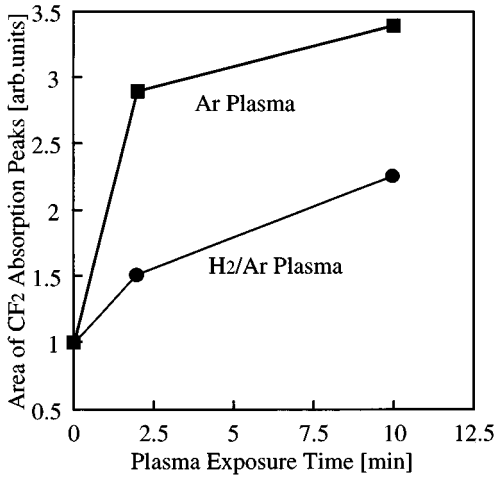

FIG. 5. The area of CF_2 absorption peak measured by *in situ* FTIR RAS as a function of Ar and H_2/Ar plasma exposure time.

density. Consequently, it is suggested that the influence of Ar* atoms on the formation of fluorocarbon films was extremely small in this experimental condition.

B. *In situ* FTIR RAS observation of the surface reaction of CF_2 radicals assisted by Ar^+ ion bombardment

First, we investigated the adsorptive reaction of CF_2 radicals on the buried metal substrate (*a*-Si surface) exposed to ECR Ar downstream plasma with CF_2 radical injection employing *in situ* FTIR RAS. Figure 4 shows FTIR spectra of (a) the fluorocarbon film formed on the *a*-Si surface in the ECR Ar downstream plasma with CF_2 radical injection; and (b) the fluorocarbon film formed on the *a*-Si surface by CF_2 radical injection without the ECR Ar downstream plasma exposure. The experimental condition was as follows: HFPO and Ar flows rate were 12.5 and 25 sccm, respectively. Total pressure was 1.3 Pa. Microwave power was 700 W. Deposition period was 5 min. In both spectra (a) and (b), the peaks of CF_2 asymmetric stretch were observed around 1160 and 1240 cm^{-1}, and the peak of CF_2 symmetric stretch was observed around 1210 cm^{-1}.[23] From these results, it is clearly observed that the injected CF_2 radicals were adsorbed on the *a*-Si surface without plasmas. By curve-fitting for each spectrum, we calculated the total area of the peaks around 1160, 1210, and 1240 cm^{-1}. As a result, the total area of the peaks in spectrum (a) was three times larger than that in spectrum (b). This fact indicates that the adsorption of CF_2 radicals on *a*-Si surface was enhanced by the assistance of ion bombardment, namely, the sticking probability of CF_2 radical on Si surface increased by the effect of ion bombardment. Moreover, the spectrum with Ar plasma exposure was observed to be broadened compared with that without Ar plasma exposure. Therefore, CF_2 radicals adsorbed on the *a*-Si surface are considered to become crosslinked by ion bombardment. This result is consistent with the results of *in situ* XPS analysis that the structure of the film became crosslinked under the ion bombardment in our previous studies.[16,17]

Next, we investigated sticking probabilities of CF_2 radicals on the fluorocarbon film surfaces with different surface structures. The CF_2-covered surfaces were formed by injecting only CF_2 radicals for 20 min on the substrate as shown in Fig. 2. The chemical structure of these samples was only CF_2 component from the result of XPS analysis. These samples were exposed by the ECR Ar or H_2/Ar downstream plasma. The plasma was generated at Ar flow rate of 25 sccm, pressure of 0.13 Pa and microwave power of 700 W. In the case of H_2/Ar plasma, H_2 and Ar flow rates were 12.5 and 12.5 sccm, respectively. The exposure time of Ar or H_2/Ar plasma was varied. The fluorocarbon films with different surface structures were formed by varying plasma exposure time. After plasma exposure, CF_2 radicals were injected again on these samples with different surface structures for 5 min. Figure 5 shows the area of CF_2 absorption peak measured by *in situ* FTIR RAS as a function of the Ar and H_2/Ar plasma exposure time. The areas of CF_2 absorption peak were normalized to unity at Ar plasma exposure time of 0 min. As shown in Fig. 5, it is observed in real time that in Ar plasma, the area of CF_2 absorption peak increased rapidly up to 2 min and was saturated with further increasing Ar plasma exposure time. The saturation-onset time of the area of CF_2 absorption peak was considered to be the time required to bombard the whole area of CF_2-covered surface by Ar^+ ions. This is confirmed by the following: We measured the saturation ion current near the substrate using Langmuir single probe method, and calculated the ion flux to the substrate. The ion flux in the Ar plasma was estimated to be 5×10^{12} cm^{-2} s^{-1}. In order to create the active sites on the whole area of CF_2-covered surface by Ar^+ ions at Ar^+ ion flux of 5×10^{12} cm^{-2} s^{-1}, assuming that the carbon density at the surface is of the order of 10^{15} cm^{-2}, Ar plasma exposure time required was estimated to be 200 s.

We investigated the change of the chemical structure of CF_2-covered surface exposed to Ar and H_2/Ar plasmas, that

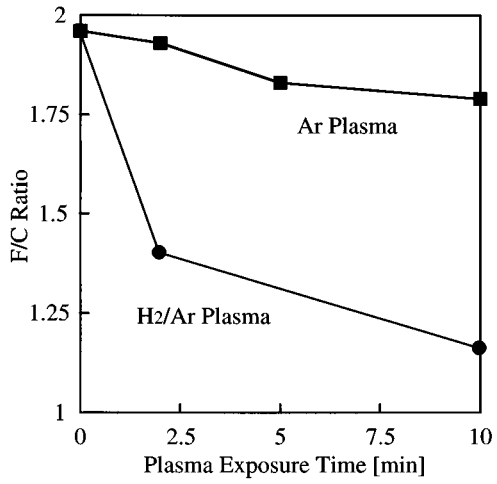

FIG. 6. F/C ratio of fluorocarbon film surface by *in situ* XPS after Ar and H_2/Ar plasmas exposure as a function of plasma exposure time.

is, the effect of ion bombardment to understand the behaviors shown in Fig. 5. Figure 6 shows the F/C ratio of the fluorocarbon film surface measured by *in situ* XPS after Ar and H_2/Ar plasmas exposure without postdeposition of fluorocarbon film as a function of the plasma exposure time. As shown in Fig. 6, the F/C ratio of films decreased with increasing plasma exposure time in the Ar plasma. This result suggests that *F* atoms on CF_2-covered surface were removed by Ar^+ ions in the ECR Ar plasma.[1] Consequently, it is confirmed that CF_2 radicals were adsorbed predominantly on the active sites formed by ion bombardment. After the whole surface of film was covered with CF_2 radicals, the sticking coefficient of CF_2 radical decreased rapidly.

As shown in Fig. 5, in the H_2/Ar plasma, the saturation-onset time of the area of CF_2 absorption peak was longer than that in the case of Ar plasma, which was due to the decrease of Ar^+ ion flux, because the Ar flow rate in H_2/Ar plasma was half of that in pure Ar plasma. The area of CF_2 absorption peak of H_2/Ar plasma in saturation region was lower than that of Ar plasma. Moreover, F/C ratio of the film in H_2/Ar plasma decreased rapidly with increasing the plasma exposure time, and was lower than that in Ar plasma as shown in Fig. 6. From these results, it was found that CF_2 radicals were not easily adsorbed on the fluorocarbon film exposed to the H_2/Ar plasma compared with the case of Ar plasma. H atoms in the H_2/Ar plasma extract F atoms from the fluorocarbon film, thereby creating active sites such as dangling bonds of C atoms on the film surface. However, other H atoms are likely to passivate the active sites of C atoms.[13] Therefore, it is considered that the active sites on the fluorocarbon film surface exposed to the H_2/Ar plasma decreased with forming of C–H bonds.

In our previous study, when CF_2 radicals were injected on the Si surface exposed to ECR H_2/Ar plasma at a microwave power of 800 W, the deposition rate of fluorocarbon film was 2.7 nm/min which was lower than that in the ECR Ar plasma (3.2 nm/min).[16] It was suggested from the results of *in situ* FTIR RAS that the lower deposition rate of fluorocarbon film in the ECR H_2/Ar plasma was caused by the decrease of the adsorptive sites of CF_2 radicals, due to H passivation of C active sites formed by Ar^+ ion bombardment.

IV. CONCLUSIONS

We clarified the mechanism of fluorocarbon film formation through CF_2 radicals in the SiO_2/Si selective etching using fluorocarbon plasma. In the fluorocarbon film formation on Si by ECR Ar downstream plasma with CF_2 radical injection, the influences of Ar^+ ions, photons, and Ar* atoms at metastable state in the ECR Ar downstream plasma were investigated. Using the permanent magnet set above the substrate in ECR Ar plasma, charged species deflected away from the substrate. Comparing the deposition rate of fluorocarbon films with and without ions, it was found that the Ar^+ ions play an important role for the fluorocarbon film formation through CF_2 radicals, and that the contribution of photons and Ar* atoms at metastable state to the fluorocarbon film formation was negligible in this study.

In situ FTIR RAS and *in situ* XPS measurements were successfully carried out to investigate the adsorptive reaction of CF_2 radicals on the surface of fluorocarbon film exposed by Ar and H_2/Ar plasmas. From these results, it was clarified that CF_2 radicals were adsorbed at a high probability on the active sites created due to the removal of *F* atoms from the fluorocarbon film surface by the bombardment of Ar^+ ions, and the adsorption of CF_2 radicals on the films exposed to H_2/Ar plasma was decreased due to the passivation of active sites by H atoms.

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