

The Effects of Plasma Treatment on a-C:F Thin Film with Low Dielectric Constant using CF₄

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Fluorinated amorphous carbon films was deposited on a p-type Si(100) substrate by using an inductively coupled plasma chemical vapor deposition(ICPCVD) system with a mixture of carbon tetrafluoride (CF₄) and methane (CH₄) gases. CF₄ plasma treatment with various treatment times was carried out in situ to the as-deposited a-C:F film, which was deposited with flow rate ratio, $R(\%) = [CF_4 / (CH_4 + CF_4)] \times 100$, of 75%. Fourier transform infrared spectroscopy and X-ray photoelectron spectroscopy spectra showed that the structure of a-C:F thin film consisted of the C-F, C-F₂ and C-F₃ bonds. The content of C-F₂ bond in plasma-treated films increased until 3 minutes, while C-F bond in the film decreased. Therefore, fluorine atomic concentration in the film increased from 61% to 65%. When CF₄ plasma applied to a-C:F thin film by 3 minutes, the dielectric constant was lowered from 2.9 to 2.65 and the refractive indices was reduced from 1.65 to 1.60. CF₄ plasma provided fluorine atoms, which reacted with the atoms in the film, and changed the properties of a-C:F film.

1. Introduction

Shrinkage of the metal-line width in ultra-large-scale integrated (ULSI) circuits needs inter-metal dielectric (IMD) layer with a low dielectric constant (low-*k*) for reducing resistance-capacitance (RC) time delay, power dissipation and cross-talking[1]. Use of present silicon dioxide (SiO₂) films as IMD layers will result in high parasitic capacitance and crosstalk interference in high-density devices[2]. Therefore it is necessary to reduce resistance and capacitance to achieve high performance. For decreasing the resistance of interconnecting metal and capacitance of the IMD layers, many researchers are studying on the copper/low-*k* based interconnect technology[3]. There are a number of

requirements for the new low dielectrics [4-6], such as low dielectric constant, high thermal stability, good electrical insulation, high mechanical strength and good adhesion to neighboring layers. Fluorinated amorphous carbon (a-C:F) film and the parylene polymer film are very good candidates for a IMD layer, because these materials have low-dielectric constant, good mechanical property and crosslinked polytetrafluoroethylene (PTFE)-like structure [6-8]. Recently, a-C:F films deposited by plasma-enhanced chemical vapor deposition (PECVD) and helicon wave plasma CVD have been considered as potential candidates for IMD materials for the next-generation ULSI circuits [6,9].

These films are deposited from fluorocarbon source gases, such as CF₄, C₂F₆, or C₃F₈, by

PECVD [6.9.10]. The a-C:F film has many advantages such as low dielectric constant, which can be changed from 2.1 to 2.8 by controlling the fluorine concentration, good gap filling performance and simplicity in process implementation. These films have the amorphous C-C crosslinked structure and related C-F_x bonds like the chemical structure of PTFE. The strong C-F_x bonds in the a-C:F film decrease its dielectric constant, while the crosslinked structure maintains the film's stability. The a-C:F films deposited by PECVD with a gaseous mixture of CF₄ and CH₄ include inevitably hydrogen atoms in the film and consist of functional groups of C-CF bonds, such as -C-CF, -CF-C-CF₂, -CF-C-CF₃, -CF-C-CF and -CF-C-CF₂ bonds, which are different from the homogeneous (CF₂)_x bond structure.

These films should have a low hydrogen concentration. Therefore, a-C:F films become hydrophobic and cause low adhesion to interconnection electrode materials [11,12].

Because the crosslinking causes the increase of adhesion, the formation of a-C:F film with the homogeneous (CF₂)_x bond structure is necessary for good adhesion. The composition and bond structure of a-C:F film can be controlled by the plasma treatment with various gases. By low pressure plasma treatment, the reactive particles and photons interact with the polymer surface. This interaction depends on the plasma condition and the nature of the polymer, and can result in the crosslinking structure [13]. Plasma contains neutral species, energetic ions, electrons, photons and many kinds of radicals. The interaction of plasma radicals with a-C:F films can give rise to chemical and physical modifications to their surface, while the bulk properties remain unchanged [14]. In this study, a-C:F films were deposited on a p-type Si(100) substrate by using an ICPCVD with a mixture of (CF₄) and (CH₄) gases. CF₄

plasma treatment with various treatment times was carried out *in situ* to the as-deposited a-C:F film. We investigated the change of characteristics in a-C:F films with CF₄ plasma treatment. The optical and electrical properties of the films were investigated and the refractive index and the dielectric constant were evaluated.

2. Experiment

The a-C:F films were deposited on a silicon substrate by using a mixture of CF₄ and CH₄ gases in a radio-frequency (13.56 MHz) ICPCVD system. The wafers were in a floating potential and not intentionally heated. The wafers were chemically cleaned by a standard cleaning procedure before loading them into the reaction chamber. The inductively coupled plasma was generated by means of a four-turn coil around a quartz tube. A base pressure was reached to 10⁻⁶ Torr and CF₄ and CH₄ gases were provided into the reaction chamber through a mass flow controller (MFC). The total flow rate kept as 40 sccm and the flow rate ratio, $R(\%) = [CF_4 / (CH_4 + CF_4)] \times 100$, was 75%. The discharge pressure was measured as about 260 mTorr by a Baratron gauge. The electron density and the electron temperature were measured by a fast injection Langmuire probe which was calibrated with a microwave interferometer. The plasma density and the electron temperature for an RF power of 800 W were about 10¹² cm⁻³ and 1.6 eV, respectively. In this discharge condition, the concentration of radicals (C, F and H) changed with the gas flow rate, so the relationship between the relative gas flow rate and film properties could be studied. To investigate the change of properties in a-C:F film by CF₄ plasma treatment, we carried out CF₄ plasma treatment with different plasma treatment times (1 minute

and 3 minutes) for an RF power of 300 W just after deposition without breaking vacuum. The CF₄ gas flow rate was fixed as 5 sccm, and the pressure for discharging was about 170 mTorr. Fourier transform infrared (FTIR, DA8 Bomem) spectroscopy, which performed in the absorbance mode, was used to determine the bonding configurations for C-F, C-F₂ and C-F₃ bonds in the films. The chemical bonding structure was characterized by using X-ray photoelectron spectroscopy (XPS, VG ESCALAB 210). The thickness and the refractive index of the a-C:F films were measured by an ellipsometer. The dielectric constant (at 1 MHz) was measured by MIS (Al/a-C:F/p-Si) structure in a vacuum to eliminate the influence of moisture.

3. Results and discussion

In order to investigate the bonding structure of a-C:F films treated by CF₄ plasma, we deconvoluted the FTIR spectra of a-C:F film by fitting the data with a number of Gaussian peaks.

Figure 1 shows the FTIR spectra of the a-C:F films prepared with different plasma treatment times. The spectra are generally broad and overlapped due to the complex stoichiometry and the amorphous nature of the films. The strong peak at 1108 cm⁻¹ is assigned to an asymmetric stretching mode of the fluorine atoms along a direction parallel to the C-F direction. The peak position is almost unchanged although the plasma treatment time increases. In the as-deposited sample, the peaks near 1214 cm⁻¹ and 1456 cm⁻¹ represent the symmetric and asymmetric C-F₂ stretching mode, respectively. Also, the weak peaks near 1306 cm⁻¹ and 1392 cm⁻¹ correspond to the C-F₃ and the CF₃-CF₂ bonds of the CF₃-CX_y stretching mode group (X_y=F on high wavenumber

side), respectively. The broad peaks in the range from 1600 cm⁻¹ to 1800 cm⁻¹ correspond to the unsaturated double bonds associated with C=C, C=CF₂ and CF=CF₂. The C=C stretching mode is normally observed at 1600 cm⁻¹ in organic compounds when C atoms are bonded to H atoms, but the peak is typically shifted to higher frequencies when the H atoms are replaced by F atoms [15].

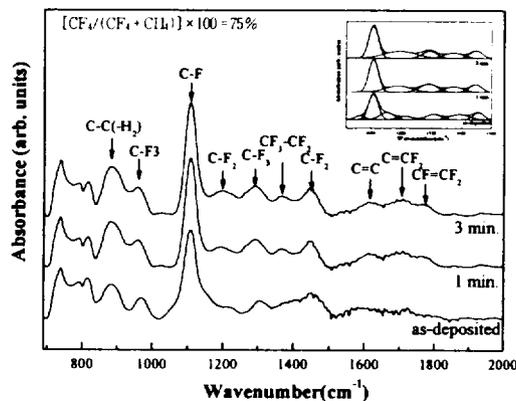


Fig. 1. The FTIR spectra of the a-C:F films as a function of plasma treatment time, when the as-deposited sample is formed with $R(\%) = [CF_4 / (CH_4 + CF_4)] \times 100$ flow rate ratio of 75%.

As plasma treatment time increases, the peak intensities of fluorine-rich function groups of C-F_x, such as C-F₂, C-F₃, CF₃-CF₂ bonds increases. The bonding structure of plasma-treated sample is similar to that of as-deposited film. But the peak position of C=C (1592 cm⁻¹) bond shifts toward higher wavenumber and that of CF=CF₂ (1782 cm⁻¹) bond shifts toward lower wavenumber after the CF₄ plasma treatment with various times. Particularly, the peaks of the symmetric and asymmetric C-F₂ stretching modes are red-shifted by 20 cm⁻¹ and 10 cm⁻¹, respectively. The peaks of C-F₃ and CF₃-CF₂ bonds are red-shifted to the

lower wavenumber of about 15 cm^{-1} and 20 cm^{-1} , respectively. It means that this frequency shifts in IR spectra are related to the changes in the bonding characteristics, such as the bonding angle and the bond length [16]. Generally, a low dielectric polymer material including F atoms has the bonding termination structure and C-F_x bonding is broken easily by thermal energy. From this result, we can infer that a-C:F film proceeds more to consist of the cross-linked structure with increasing the C-F₂ bond by fluorination of film surface, since some of the carbon atom in the surface layer of the a-C:F film were broken with fluorine atom by fluorine ion species during the post-plasma treatment process, and fluorine atoms adsorbed to the film surface from CF₄ plasma [17].

Figure 2 shows the relative integrated absorption areas of C-F, C-F₂ and C-F₃ peaks calculated from the results of fitted peaks in Fig. 1 as a function of plasma treatment time. In Fig. 2, the integrated absorption area of C-F bond decreases slightly from 40% to 39% in case of 1 minute CF₄ plasma treatment and decreases from 40% to 37% for 3 minutes. On the other hand, the relative areas of the C-F₂ and C-F₃ bonds increase when the plasma treatment time is 1 minute. For plasma treatment of 3 minutes, the relative area of C-F₃ bond decrease to 25% from 27% of as-deposited case. The integrated absorption area of the related C-F₂ bonding components changed abruptly during the plasma treatment, while that of C-F₂ bond was increased to 38%. In the glow discharge condition, CF₄ plasma includes reactive atoms or radicals such as F, C-F, C-F₂ and C-F₃. From these results, we know that the composition of a-C:F film proceeds to the homogeneous (C-F₂)_x bond structure with the reduction of the relative areas of C-F and C-F₃ bonds. It means that the bonding structure of the a-C:F film is changed to the homogeneous C-F₂ due to the

surface fluorination by plasma treatment and is likely to form the cross-linked structure [18]. The cross-linked structure in the film was enhanced and the bonding structure of the film changed from C-F bond to the fluorine-rich functional groups of the C-F_x bond attached at skeleton chains. Therefore, we can infer that the cross-linked structure in the film was enhanced and the bonding structure of the film rearranged from C-F bond to the fluorine-rich functional groups of the C-F_x bond attached at skeleton chains, and the film surface changed to become hydrophobic by the C-F_x bond [14].

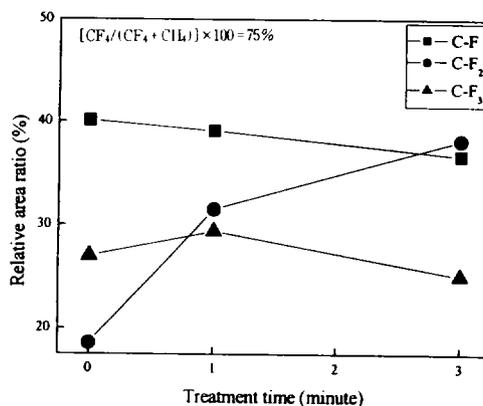


Fig. 2. The integrated absorption areas of the C-F, C-F₂, C-F₃ bonds calculated from the fitting of Fig. 1 as a function of plasma treatment time.

To confirm the existence of the related C-F_x bonds in the film surface, we deconvoluted the XPS narrow scan spectra of the C 1s peak by fitting the data with a number of Gaussian peaks. The C 1s spectra are interpreted by the chemical shift assignments from the previous results [12,19,20]. The XPS C 1s electron orbital spectra of as-deposited films with the flow rate ratio of 75% and the samples treated by CF₄ plasma with various plasma treatment times are shown in Fig. 3. The

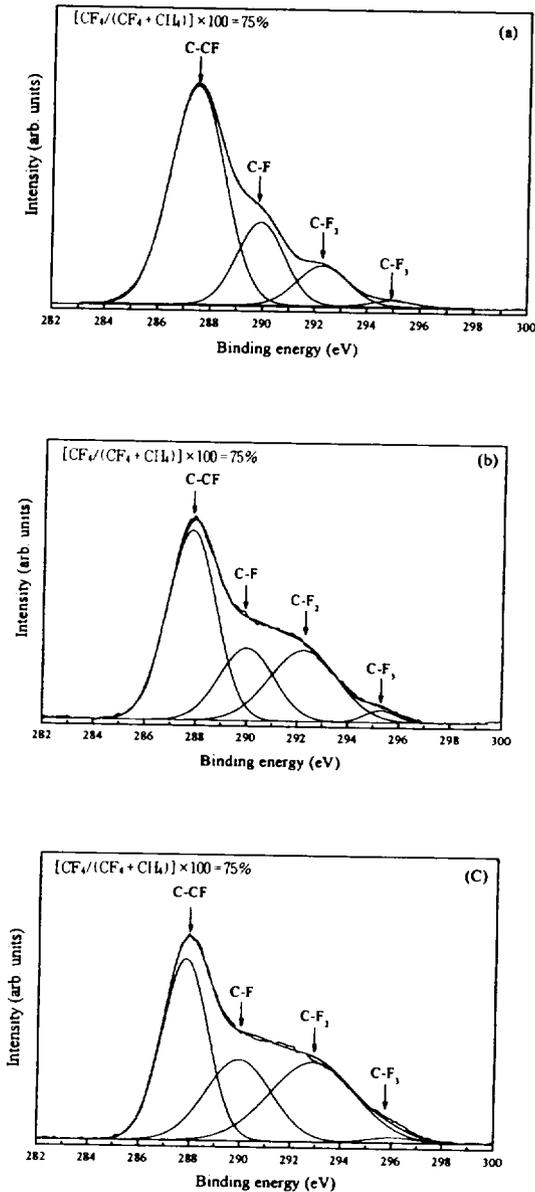


Fig. 3. C1s electron orbital XPS narrow spectra of the a-C:F films with various plasma treatment times: (a) as-deposited film. (b) film treated for 1 minute. (c) film treated for 3 minutes.

peaks were deconvoluted into four different chemical components such as: the C-CF, C-F, C-F₂ and C-F₃ bonds to consist of a-C:F film, and the

major peak was assigned to C-CF bond. Figure 3(a) shows the spectrum of as-deposited film. The four peaks were assigned such as C-CF at 287.4 eV, C-F at 289.9 eV, C-F₂ at 292.3 eV and C-F₃ at 294.9 eV, which the C-F, the C-F₂, and the C-F₃ the can be assigned the shoulder of the C-CF bond. The relative area of the C-CF bond is 65.5%, the C-F bond is 21.6%, the C-F₂ bond is 11.5% and the C-F₃ bond is 1.4%. Therefore, the C-CF bond of as-deposited films can consist of carbon-attached to functional groups of -C-CF-, CF-C-CF₃, -CF-C-CF and -CF-C-CF₂ chain structure[19]. Figure 3(b) shows the spectrum of the film treated by CF₄ plasma for 1 minute, the four peaks is assigned such as C-CF at 287.8 eV, C-F at 290.0 eV, C-F₂ at 292.2 eV and C-F₃ at 295.3 eV. The peaks of the C-CF and C-F₃ bonds were shifted about 0.4 eV to higher binding energy than that of as-deposited film. The binding energy of the C-F and C-F₂ bonds are similar that of as-deposited film. The relative area of the C-CF bond is 50.5%, the C-F bond is 21.3%, the C-F₂ bond is 26% and the C-F₃ bond is 2.2%. The relative areas of the C-F₂ and C-F₃ bonds increased about 15% and 0.8% than that of as-deposited film, and the relative areas of the C-CF bond decreased about 15% than that of as-deposited film. The C-F bond is not changed.

Figure 3(c) shows the spectrum of the film treated by CF₄ plasma for 3 minute in which the peaks were assigned into following components such as C-CF at 287.8 eV, C-F at 289.9 eV, C-F₂ at 292.9 eV, and C-F₃ at 296.0 eV. The binding energy of the C-F₂ bond shifts to higher binding energy about 0.4 eV than that of as-deposited film and the peaks of the C-F₂ bond and the C-F₃ bond shift to higher binding energy about 0.6 eV and 1.1eV, respectively. The binding energy of the C-F bond is not changed. The relative area of the C-CF, C-F, C-F₂, and C-F₃ bonds are about

40.9%, 25.4%, 32.5%, and 1.2% respectively. The relative area of the C-CF bond decreased about 9.6% than that of the film treated by CF_4 plasma for 1 minute, and the relative area of the C-F and C-F₂ bonds increased about 4.1% and 6.5% than that of the film treated by CF_4 plasma for 1 minute. These results mean that for a-C:F films as CF_4 plasma treatment time increases experienced a re-arrangement of the chemical bonds in the film: the carbon-attached to functional groups of -C-CF-, CF-C-CF₂-, -CF-C-CF and -CF-C-CF₂ bonds, change to the fluorine-rich functional groups of CF_x such as -CF₂-, CF₂-CF- and -CF₂-CF₂- chain structure. Therefore, we can infer that CF_4 plasma treatment reduces the content of the C-CF bond and increases the number of C-F₂ bonds in the film. A greater incorporation of fluorine bonded with carbon is associated with the shift toward higher binding energy due to the highest electronegativity of fluorine [21]. Therefore, the higher binding energy peak in the XPS spectrum can be assigned to more fluorine-incorporated bonding configurations. This chemical shift of the C-CF binding energy is resulted from fluorine atoms, which have the highest electronegativity, 4.0 eV, attached to the carbon atom. As referred above, we suggest that an increase of the C-F₂ bond is due to the adsorption of fluorine atom in the film surface.

Figure 4 shows the refractive index of the a-C:F films with various CF_4 plasma treatment time. The refractive index of the a-C:F films is lowered from 1.65 to 1.63 when the plasma treatment applied to the thin film for 1 minutes, and of a-C:F film with the treatment time of 3 minutes is 1.60. As plasma treatment time increases the refractive index is reduced gradually. The refractive index is related to the electronic dielectric constant, such as $n = \sqrt{\epsilon_e}$ and the electronic polarization, P_e is given by $P_e = \epsilon_0(\epsilon_e - 1)E$. Where $\epsilon_0(\epsilon_e - 1)$ represents the electronic susceptibility, χ_e . In the as-deposited film, the electronic susceptibility is calculated from $1.524 \times 10^{-11} (\text{C/v} \cdot \text{m})$. χ_e of the film treated by CF_4 plasma for 1 minute experiences χ_e to decrease from $1.524 \times 10^{-11} (\text{C/v} \cdot \text{m})$ to $1.466 \times 10^{-11} (\text{C/v} \cdot \text{m})$. When a-C:F film is treated for 3 minutes by CF_4 plasma, χ_e decreases from $1.524 \times 10^{-11} (\text{C/v} \cdot \text{m})$ to $1.381 \times 10^{-11} (\text{C/v} \cdot \text{m})$. From these results, we knew that the a-C:F films with CF_4 plasma treatment have lowering dielectric constant because the susceptibility is related to the dielectric constant by $\chi_e = \epsilon_e - 1$, which the plasma treatment films is re-arranged on the surface layer due to the fluorine-rich functional groups of CF_x bonds.

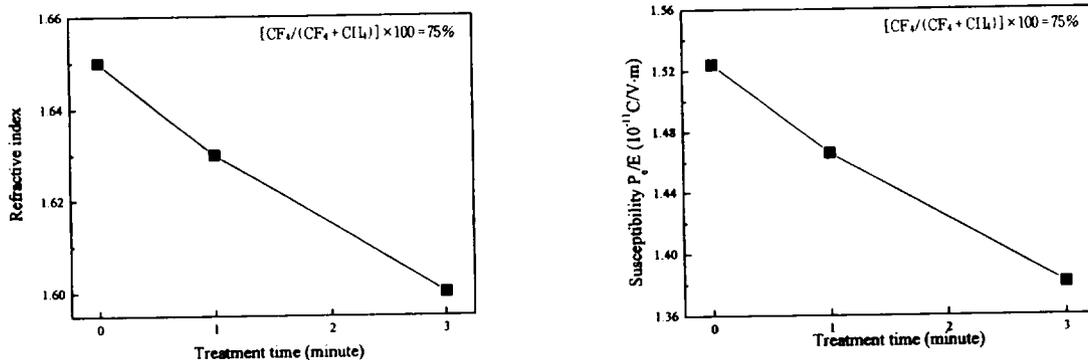


Fig. 4. The optical property of a-C:F films treated by CF_4 plasma with various times: (a) refractive index and (b) electronic susceptibility.

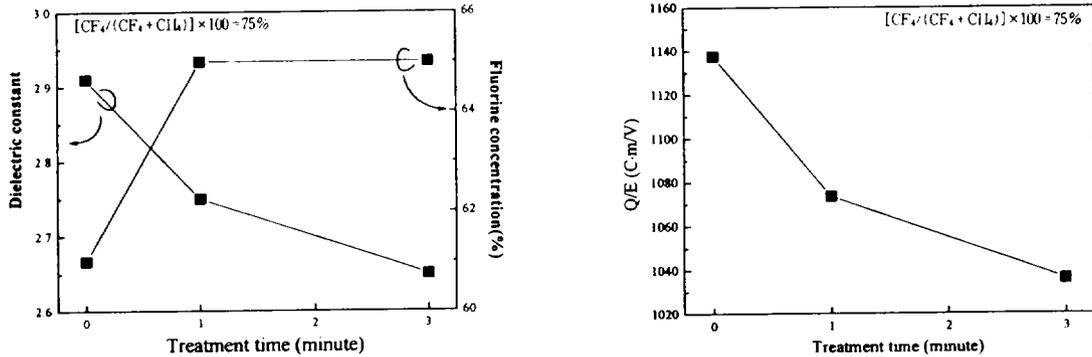


Fig. 5. (a) The F atomic concentration and the dielectric constant and (b) the surface charge of the a-C:F films as a function of plasma treatment time.

Figure 5(a) shows the fluorine atomic concentration and the dielectric constant of the a-C:F films versus plasma treatment time. The fluorine concentration and dielectric constant of as-deposited film are 61% and 2.91, respectively. At the treatment time of 1 minute, the fluorine concentration increase to 65% and the dielectric constant decrease to 2.75. However, when as-deposited film is treated for 3 minutes with CF₄ plasma, the fluorine concentration has the same value of 65% with the sample treated for 1 minute and the dielectric constant is reduced to 2.65. This can be attributed to the concentration of the the C-F₂ bond in the film. The multiplication of the capacitance, C , and the film thickness, d , is appeared to the surface charge per electric field, Q/E , and the dielectric constant is proportional to the charge. These relations are written by $Cd = Q/E = \epsilon A$ Where, ϵ is the permittivity of dielectric material, E is the electric field and A is the electrode area. Figure 5(b) shows the surface charge per the electric field which applies to the a-C:F thin film. The film surface charge per electric field is lowered from 1137.5 (C·m/V) to 1073.6 (C·m/V) when a-C:F film is treated by CF₄ plasma for 1 minute. When the film is treated by CF₄ plasma

Table 1. The electrical property and the fluorine atomic concentration as a function of plasma treatment time.

	As-deposited	1 min.	3 min.
Fluorine Atomic Concentration	61	65	65
Pe/E (10 ⁻¹¹ C/Vm)	1.524	1.466	1.381
Q/E (C ² m/V)	1137.5	1073.6	1036.2
Dielectric constant	2.9	2.75	2.65

Pe/E : Electronic susceptibility.

Q/E : Film surface charge per electric field

for 3 minutes, the film surface charge per the electric field is reduced from 1137.5 (C·m/V) to 1036.2 (C·m/V). These mean that a-C:F thin film have the lower surface charge as plasma treatment time increases. Above all, The film thickness is reduced about 8.6% to treat with CF₄ plasma for 1 minute and is reduced about 10.3% to treat with CF₄ plasma for 3 minutes but the capacitance of the plasma-treated film is unchanged almost. These facts imply that the dielectric constant of the films treated by CF₄ plasma is reduced because of the susceptibility reduction to decrease the polarizability. Therefore, we suggest that the a-C:F thin film treated by CF₄ plasma is denser than as-deposited film with forming the

C-F₂ bonding structure in the film, and the C-F₂ bond in a-C:F thin film plays a role to decrease the surface charge and the dielectric constant of the thin film denses.

4. Conclusion

The a-C:F thin film was deposited by ICPCVD without intentional heating or biasing, with flow rate ratio, $[\text{CF}_4/(\text{CH}_4 + \text{CF}_4)] \times 100$, of 75%. CF₄ plasma was applied to the as-deposited a-C:F thin film with various times. The as-deposited a-C:F film had the C-F bond in addition to C-F₂, C-F₃, CF₃-CF₂, C=CF₂ and CF=CF₂ bonds, and the chain structure of the a-C:F film can consist of the fluorine-rich functional groups of C-CF bonds, such as -C-CF, CF-C-CF₃, -CF-C-CF and -CF-C-CF₂ bonds. As the plasma treatment time increases, the number of the C-F₂ bonds increases and that of the C-CF bonds decreases. Then the bonding structure of a-C:F thin film changes to the homogeneous C-F₂ bonds by plasma treatment. The increase of C-F₂ bond by CF₄ plasma treatment reduce the refractive index and the dielectric constant of a-C:F film. The refractive index of a-C:F thin film decreased 1.65 to 1.60 by CF₄ plasma treatment of 3 minutes. In same case, the dielectric constant of the plasma-treated sample decreases from 2.9 to 2.65 and the fluorine atomic concentration increases from 61% to 65%. From these results, we can infer that CF₄ plasma treatment induces more homogeneous C-F₂ bonding structure of the a-C:F film and improve the dielectric and optical properties of a-C:F film, since more fluorine atoms are adsorbed to the film surface from CF₄ plasma during the post-plasma treatment process. And the CF₄ plasma treatment decreases the electronic polarization and the surface charge of

the a-C:F thin film and makes the a-C:F thin film dense.

Acknowledgments

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저유전 상수를 갖는 a-C:F 박막에 CF₄ 플라즈마 처리효과

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a-C:F 박막은 CF₄와 CH₄로 혼합된 가스를 사용하여 유도결합 플라즈마 화학기상증착방법으로 p-형 Si(100) 기판에서 증착하였다. CF₄ 플라즈마 처리는 $R(\%) = [CF_4 / (CH_4 + CF_4)] \times 100$ 의 유량비가 75%로 하여 실온에서 증착된 박막을 시간변화에 따라 수행하였다. C-F, C-F₂ 그리고 C-F₃ 결합으로 구성된 a-C:F 박막의 결합구조는 FTIR 과 XPS 스펙트럼으로 조사하였다. 플라즈마로 처리된 박막에서 C-F₂ 결합의 농도는 처리시간이 3분까지 증가하였고, C-F 결합의 농도는 감소하였다. 따라서 박막에서 F의 농도는 61%에서 65%로 증가하였다. a-C:F 박막을 3분 동안 CF₄ 플라즈마로 처리했을 때 유전상수는 2.9에서 2.65로 감소되었고, 굴절률은 1.65에서 1.60으로 감소되었다. 이것은 CF₄ 플라즈마 처리가 F 원자를 공급하여 박막내에서 반응에 의한 a-C:F 박막의 물성을 변화시킨다.