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The deposition of SiOF film with low dielectric constant in a helicon plasma source

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SiOF films deposited by a helicon wave plasma chemical vapor deposition method has been characterized using Fourier transform infrared spectroscopy and ellipsometry. High density plasma of $>10^{12}$ cm^{-3} can be obtained on a substrate at low pressure ($<$10 mTorr) with rf power $>$400 W with a helicon plasma source. A gas mixture of SiF_4, O_2, and Ar was used to deposit SiOF films on 5 in. Si(100) wafers not intentionally heated. Optical emission spectroscopy was used to study the relation between the relative densities of the radicals and the deposition mechanism. It was found that the addition of Ar gas to the SiF_4/O_2 mixture greatly increased the F concentration in the SiOF film. Discharge conditions such as gas composition, sheath potential, and the relative densities of the radicals affect the properties of the film. The dielectric constant of the SiOF film deposited using the helicon plasma source was 3.1, a value lower than that of the oxide film by other methods.
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Interlayer dielectric film formation technology is essential for the fabrication of multilevel interconnections for the ultralarge scale integrated circuit (ULSI). Low temperature deposition is required for multilevel interconnection interlayer dielectrics because thermal stress affects device characteristics and wiring reliability. Low temperature deposition techniques using organic sources such as tetraethylorthosilicate (TEOS)-ozone atmospheric pressure chemical vapor deposition (APCVD), H_2O-TEOS plasma-enhanced chemical vapor deposition (PECVD), biased electron cyclotron resonance (ECR) chemical vapor deposition (CVD), and helicon wave plasma CVD have been investigated for this purpose.[1–4] Helicon wave plasmas[5,6] have potentially attractive features in plasma applications because of an efficient high density plasma production compared with other conventional type plasma sources.[7,8]

In this letter we present the results on SiOF film deposition in helicon discharges at 13.56 MHz fed with SiF_4-O_2 mixtures, obtained by changing rf power, total pressure, and SiF_4/O_2 flow ratio. The system investigated here has been characterized by optical emission spectroscopy (OES) to determine the relative density of radical species, and a Langmuir probe was used to determine microscopic plasma parameters such as electron density and electron temperature. The film qualities such as dielectric constant, bonding mode, and F concentration are investigated as functions of the plasma parameters.

The SiOF film deposition is done on p(100) 5 in. silicon substrates with SiF_4 and O_2 gases, and the addition of Ar gas. The wafers are left at floating potential and not intentionally heated. The deposition of the SiOF film is carried out as a function of the SiF_4/O_2 flow rate ratio and rf power. Typical variable settings of the deposition process are an rf power of 1.4 kW and a magnetic field strength of 707 G. Such film properties as refractive index and chemical bonding structure are investigated. Fourier transform infrared (FTIR) spectroscopy, performed in absorbance mode with a model DA8 Bomem spectrometer, is used to determine the Si–F and Si–O bonding configurations in the films. The thickness and refractive index of the deposited SiOF films are measured using an ellipsometer. Electrical properties such as dielectric constant (1 MHz) are also investigated using MIS(Al/0.4 μm thick film/p-Si) structure. Optical emission spectroscopy is used to study the relation between the relative concentrations of the radical (F, Si, and O) species and the deposition mechanism. The optical emission spectroscope is set up with a high gain diode array (OMA III from EG&G Applied Research) attached to a triple grating monochromator that detects the emission of the excited species in the plasma through a quartz window set on the reactor sidewall. The Ar gas is used to study the characteristics of the helicon plasma. Such plasma parameters as electron density and electron temperature are investigated as a function of the rf power in Ar discharge.

The dependence of plasma density on rf power, obtained at $p=3$ mTorr for various magnetic field strengths, is shown in Fig. 1. The plasma density was measured at the downstream region (reaction region) ~50 cm away from the plasma source region where the helicon antenna was set. Above 530 Gauss, there is a steep density increase around 400 W of rf power. That is, it is observable that the minimum rf power required to excite helicon waves exists for different magnetic fields. The phenomena of the existence of a threshold rf power needed to excite helicon waves agrees with Chen and Chevalier's[9] and Shoji *et al.*'s results.[10] A helicon wave cannot be excited unless the plasma density is above a

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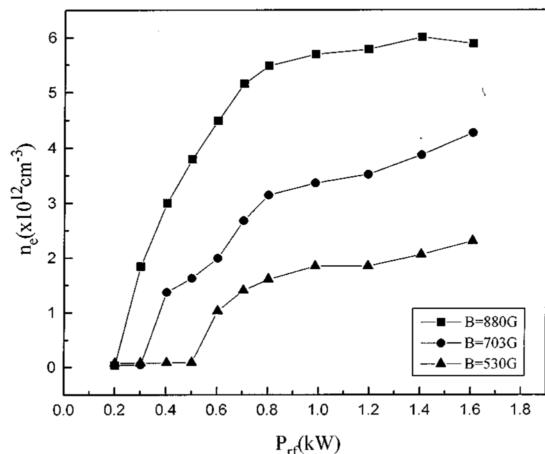

FIG. 1. Dependence of Ar plasma density on rf power at 3 mTorr for different magnetic field strengths.

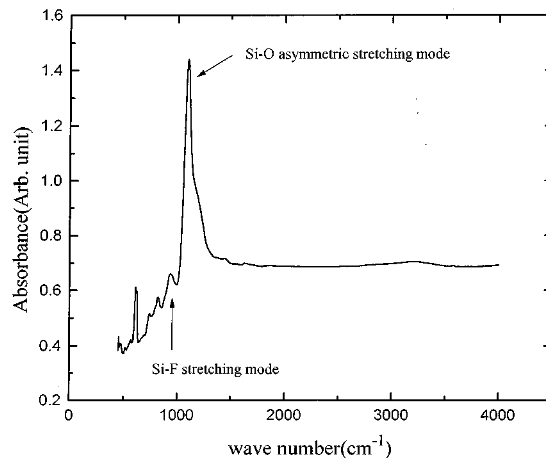

FIG. 3. The infrared spectrum of SiOF film.

critical density satisfying the dispersion relation of the helicon wave.[9,10] A minimum rf power for helicon wave plasma production can be lowered by increasing the magnetic field strength due to the confinement effect of the magnetic field. The electron density becomes saturated with different values for different magnetic fields, respectively, above 1 kW. The saturation density in the downstream region increases as the magnetic field increases, since the radial plasma loss decreases as the magnetic field increases.

The emission intensities of F(703 nm), Si(728 nm), and O(777 nm) species normalized by the ion saturation current density are plotted as a function of rf power in Fig. 2. In the helicon mode (above rf power of 1000 W), SiF_4 is well dissociated into Si and F atoms. In the mixture of Si_4 and O_2 gases, the threshold rf power for the helicon mode is higher than that in the Ar discharge. Since helicon wave plasma has a hot electron tail and high density electrons,[11,12] SiF_4 is very efficiently dissociated into Si and F atoms. The deposition rate as a function of rf power is also shown in Fig. 2. When the discharge is not in the helicon mode below threshold rf power, no deposition is observed. In this case, the emission intensities of the F and Si lines are much lower than they are

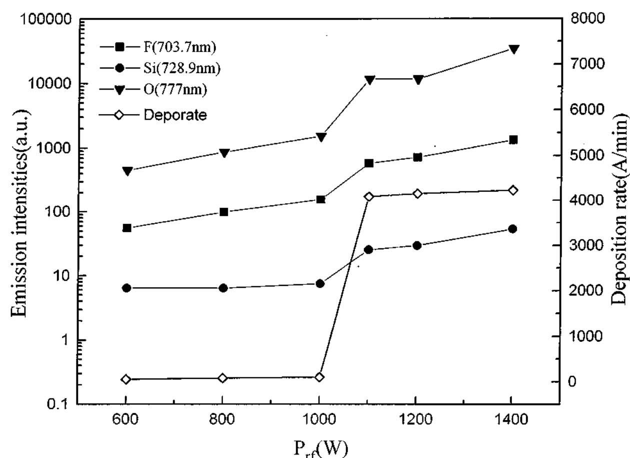

FIG. 2. Emission intensities of F, Si, and O atoms normalized by the ion saturation current density.

in the helicon mode. The deposition rate is measured to be nearly constant as the Si_4 and O_2 flow rate ratio changes in the helicon mode. It has been found that the deposition rate strongly depends on the degree of dissociation of the Si_4 molecules into Si and F atoms.

The infrared spectrum of SiOF film is shown in Fig. 3. The absorption peaks corresponding to Si–O and Si–F bonds are at around 1090 and 930 cm^{-1}, respectively. The peak corresponding to OH is not observed because a gas source containing H is not used. The absence of any measurable OH content in the oxide film is an indicator of a decrease in porosity.[13] The peak positions of the Si–O–Si stretching mode is higher than that of thermal oxide or the oxides of other methods by about 30–50 cm^{-1}. The peak position of the Si–O–Si stretching mode increases as the SiF_4/O_2 flow rate ratio increases as shown in Fig. 4(a). Changes in position and FWHM of the Si–O–Si asymmetries stretching mode peak can indicate changes in stoichiometry, density, porosity, composition, strain, and structure.[14–17] An increase in the main peak position and a decrease in the FWHM indicate a lower porosity in deposited films as compared to thermal silicon dioxide.[16]

The normalized emission intensities of the F, Si, and O species as a function of the SiF_4/O_2 flow rate ratio are shown in Fig. 4(a). As the fraction of Si_4 increases, the relative densities of the F and Si species increase and the relative density of O atom slowly decreases. Therefore, the concentration of Si and F in the gas phase seems to be a limiting factor determining the qualities of the film. The concentration of Si and F in the gas phase can be controlled by changing the flow rate ratio, operating pressure, rf power, and the magnetic field strengths. The dependence of the dielectric constants on the SiF_4 flow rate is shown in Fig. 4(b). The dielectric constant of the SiOF films decreases as the fraction of SiF_4 flow rate increases. The dielectric constant of the sample obtained at the flow rate of $SiF_4/O_2=5$ sccm/5 sccm was measured as 3.1. In this sample, the concentration of F atom analyzed by ESCA is 21%. On the other hand, the concentrations of F atom in samples obtained at other conditions are about 13%. It seems that the concentration of F in the SiOF film is an important factor determining the optical and electrical properties of the films. The silicon atom

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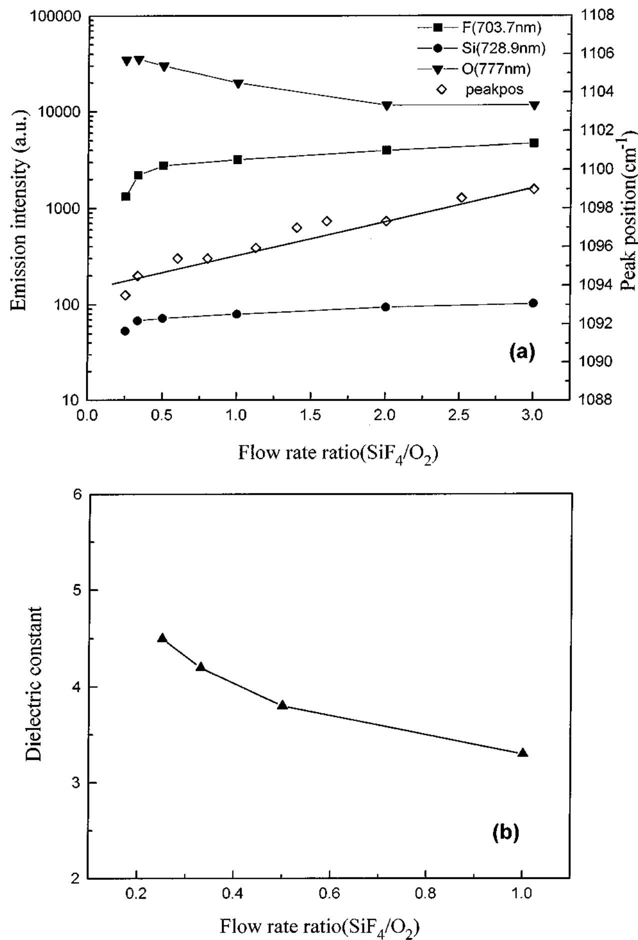

FIG. 4. (a) The peak positions of the Si–O–Si stretching bonding mode and the emission intensities of F, Si, and O as a function of the SiF_4/O_2 flow rate ratio. (b) The dependence of the dielectric constant of the SiOF films.

bonded with the fluorine atom has a slight positive charge because of the large electronegativity difference between fluorine and silicon. Therefore, the dielectric constant of the SiOF film may depend on the concentration of F atom in the film and is lower than that of the thermal oxide.

When Ar gas is added to the SiF_4/O_2 mixtures, the concentration of F in the SiOF film increases. Since negative F^- and O^- ions can be highly produced in the absence of Ar gas, the plasma potential becomes lower. Thus, the sheath potential is also lowered, and the energy of the positive ion incident on the substrate is lowered. If Ar gas is added to the Si_4 and O_2 mixtures, the density of the positive Ar ion becomes high. Therefore, the plasma potential (or sheath potential) becomes higher in comparison with the absence of Ar gas. Hence, the positive Ar ions incident on the substrate with higher energy may have an affect on the formation of the Si–F bond on the surface reaction. It was reported that the dielectric constant of SiOF film deposited by the rf biased ECR CVD method decreases as the rf bias increases.[18] It is suggested that the F concentration in the SiOF can be controlled by changing the gas composition and such plasma parameters as plasma potential and ion energy. A detailed analysis of the effect of the F fraction on the optical and electrical properties of the films will be presented in future publications.

In conclusion, a fully ionized plasma is produced by more than threshold rf power at low pressure (<10 mTorr) in the helicon plasma source. In the condition of the helicon mode, the Si_4 and O_2 molecules are efficiently dissociated and the deposition of SiOF film on 5 in. Si(100) wafers not intentionally heated is performed with ~400 nm/min of the deposition rate. The concentrations of Si and F in the gas phase seem to be a limiting factor determining the quality of the film. SiOF film of low dielectric constant, about 3.1, can be obtained by changing the gas composition, rf power, and magnetic field. The addition of Ar gas to the SiF_4-O_2 mixture causes a great increase in the percentage of atomic F in the SiOF film. It is suggested that the helicon plasma source has an attractive advantage for the application of SiOF film deposition because of the high dissociation rate and high plasma density associated with the hot electron tail.

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