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Effect of DC Bias Voltage on the Characteristics of Low Temperature Silicon–Nitride Films Deposited by Internal Linear Antenna Inductively Coupled Plasma Source

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The characteristics of silicon–nitride deposited at the temperature lower than 100 °C using an internal linear-type inductively coupled plasma were investigated as functions of the NH₃/SiH₄ ratio and ion bombardment energy applied to the substrate (dc bias voltage) for use as the gate dielectric material of flexible display devices. Decreasing the NH₃/SiH₄ ratio to 2 and increasing the dc bias voltage to –150 V decreased the Si–O bonding and increased the Si–N bonding, resulting in a more nitrogen-rich SiN_x thin film. In addition, the capacitance-voltage measurement of the metal-insulator-semiconductor devices fabricated with the SiN_x thin film deposited at various dc bias voltages showed a hysteresis curve in the cyclic voltage measurement and the increase of the dc bias voltage with decreasing hysteresis voltage. The interface trap density measured at a dc bias voltage of –150 V and NH₃/SiH₄ ratio of 2 showed the lowest interface trap charge density of about 2 × 10¹¹ cm⁻². Under these conditions, the dielectric constant was as high as 7.2. © 2010 The Japan Society of Applied Physics

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1. Introduction

Silicon–nitride (SiN) films have been widely used in various important applications ranging from semiconductor devices to flat panel displays, such as passivation layers for diverse microelectronics, gate dielectric and optical materials for thin film transistors (TFTs), mask layers for silicon KOH etching,¹⁾ anti-reflection (AR) coatings for solar cells,²⁾ etc. Especially, due to their chemical inertness, excellent dielectric properties, and thermal stability, silicon nitride films have been studied for several decades as one of the gate dielectric materials which could replace silicon oxide in metal-oxide-semiconductor device technology and are currently used as the gate dielectric material for TFTs for flat panel display devices.

Many researchers have attempted to develop high quality silicon nitride films using various deposition methods. The main deposition technique used to obtain high quality silicon nitride films was low pressure chemical vapor deposition (LPCVD) using a mixture of reactant gases consisting of SiH₄ and N₂ or NH₃.³⁾ In this case, film formation takes place as a result of the reactions between a number of exited species such as N, NH_x, and SiH_x.⁴⁾ In addition, by the application of a plasma during the CVD, that is, by using plasma enhanced CVD (PECVD), silicon nitride can be deposited at a lower temperature (~300 °C),^{5–7)} whereas it is deposited at a temperature of ~800 °C in the case of LPCVD.⁸⁾ However, the deposition of silicon nitride at a lower temperature by using conventional PECVD shows some problems, such as poor nitride/Si interface properties and a high bulk trap density in the silicon nitride film, causing the degradation of the electric properties of the devices, in addition to the presence of hydrogen in the film.⁹⁾

These days, organic devices such as organic TFTs (OTFTs) and organic light emitting diodes deposited on glass substrates and flexible display devices are being actively investigated as the next generation display devices. For these devices, even though some organic gate dielectrics are being investigated, the reliable inorganic gate dielectrics, such as the silicon nitride used in current TFTs, are being more actively investigated. In order for it to be used in these devices, silicon nitride deposited at a temperature lower than 100 °C is re-

quired. To deposit silicon nitride at a temperature lower than 100 °C while retaining a high deposition rate and high quality thin film, high density plasma sources, such as inductively coupled plasmas (ICPs), electron cyclotron resonance (ECR), etc., have also been utilized.^{10,11)} Especially, among the various high density plasma sources, ICP sources have been most widely used, due to their relatively simple source structure, in addition to their easier scalability to a larger area.^{12,13)} In addition, as the source size becomes larger than a meter, the standing wave effect and capacitive coupling to the plasma became more significant. Therefore, in order to overcome these problems, many researchers have studied internal type ICP sources with various ICP source antenna configurations. For example, in the case of Wu *et al.*,¹⁴⁾ a traveling wave was introduced to overcome the standing wave problem caused by the long internal antenna length connected in series as well as to obtain uniform plasmas. In the case of Setsuhara *et al.*,¹⁵⁾ the low-inductance internal antenna configuration was used to suppress the electrostatic coupling and to obtain the high density plasma. However, even with the ICP-PECVDs, it is still difficult to obtain both high quality silicon nitride thin films required for the gate dielectrics at the deposition temperatures lower than 100 °C.

In this study, to obtain high quality silicon nitride thin films at a low temperature (<100 °C), the dc biasing of the substrate was utilized during the deposition of the SiN_x thin film with an ICP source. For the extension of the ICP source to a larger area, an internal linear-type ICP, which is extendable to sizes larger than 2 m,¹⁶⁾ was used with the reactant gases (NH₃/SiH₄) and the effects of the ratio of NH₃ to SiH₄ and dc biasing on the properties of the thin film were investigated.

2. Experimental Methods

A schematic diagram of the experimental setup consisting of an ICP-PECVD system with an internal linear U-type antenna used in this study is shown in Fig. 1(a). A square shaped substrate with dimensions of 175 × 175 mm² was installed in the cylindrical processing chamber having a diameter of 380 mm, and a U-type antenna module consisted of two internal linear antennas connected to each other (total length of the U-type antenna was 1650 mm and the distance between two linear antennas was 250 mm) was installed

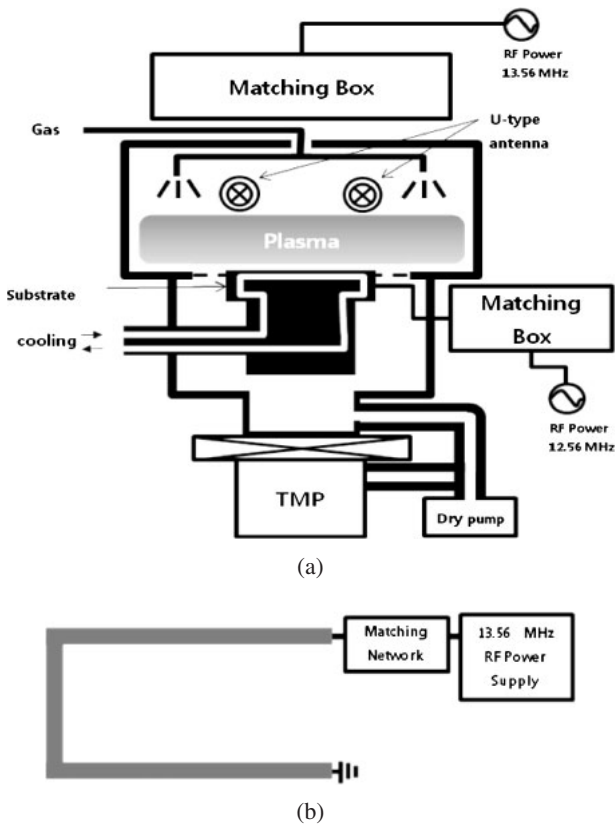


Fig. 1. (a) Schematic diagram of the linear internal-type ICP system used in this experiment. (b) Configuration of the internal-type ICP antenna connection.

above the processing chamber. The inner conductor of each antenna was made of copper tube (10 mm in diameter) and was shielded by a quartz dielectric tube (33 mm diameter) to isolate it from the plasma. For the cooling of the antenna, water was flowed through the copper tube. The distance between the U-type antenna and the substrate holder was 150 mm. As shown in Fig. 1(b), one side of the U-type antenna was connected to a 13.56 MHz rf power supply through an L-type matching network and the other side of the antenna was connected to the ground. Also, a 12.56 MHz rf power supply was used to deliver the power to the substrate for the formation of the dc bias voltage.

A gas mixture consisting of $\text{NH}_3/\text{SiH}_4/\text{Ar}$ was fed to the chamber from the top of the chamber, where SiH_4 and NH_3 gases were used as the reaction gases and Ar gas as the ignition gas. The SiN_x films were deposited on p-type (100) silicon substrates. The total gas flow rate of the $\text{NH}_3/\text{SiH}_4/\text{Ar}$ gas mixture was maintained at 110 sccm and the operating pressure at 10 mTorr. The 13.56 MHz rf power supplied to the ICP source and the dc bias voltage supplied to the substrate by the application of the 12.56 MHz rf power were varied from 300 to 1000 W and from 0 to -150 V, respectively. While depositing the SiN_x thin film, the substrate temperature was increased slowly with the increase of processing time due to the exposure to the plasma and ion bombardment, however, due to the cooling of the substrate holder during the processing, the substrate temperature could be maintained at the temperature below 100°C for all of the experimental conditions.

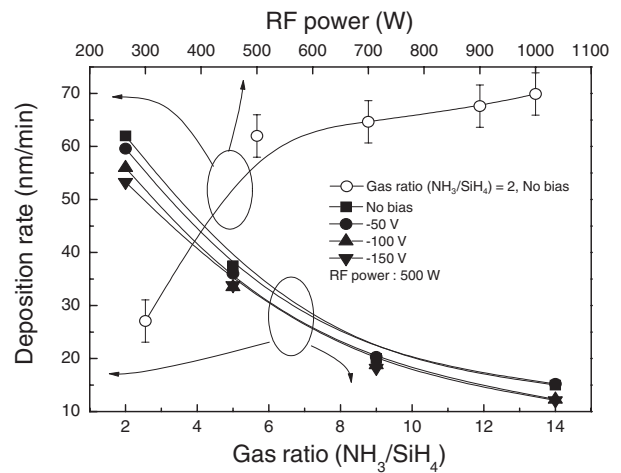


Fig. 2. Deposition rate as functions of the NH_3/SiH_4 gas ratio (at an rf ICP source power of 500 W) and 13.56 MHz rf ICP source power (at a gas mixture ratio of $\text{NH}_3/\text{SiH}_4 = 2$).

The compositions, binding states and refractive indices of the SiN_x thin films were measured using a Fourier transform infrared spectrometer (FTIR; Bruker IFS-66/S) and an ellipsometer (J. A. Woollam M-2000, VASE Manager) at a wavelength of 632.8 nm, respectively. The ratio of Si/N in the deposited thin film was also investigated using X-ray photoelectron spectroscopy (XPS; Thermo VG MultiLab 2000, Mg $K\alpha$ source). In addition, metal/insulator/semiconductor (MIS) capacitors having the structure of Al/insulator (deposited SiN_x)/p-Si were fabricated and their flat-band voltage and hysteresis voltage at a high frequency (1 MHz) were measured by the capacitance–voltage ($C-V$) method. The thicknesses of the Al layer and SiN_x of the MIS capacitors were maintained at 100 and 300 nm, respectively. The $C-V$ curve measurement system consisted of a switch control unit (HP 3488A), voltage source (Keithley 230), capacitance meter (Boonton 17200) and programmable electrometer (Keithley 617). A step profilometer (Tencor Alphastep 500) was used to measure and adjust the thickness of the film.

3. Results and Discussion

Figure 2 shows the effect of the NH_3/SiH_4 ratio and rf power to the ICP source on the deposition rate of SiN_x . The operating pressure was maintained at 10 mTorr by keeping the Ar gas flow rate at 50 sccm and the flow rate of $\text{NH}_3 + \text{SiH}_4$ at 60 sccm. When the gas ratio of NH_3/SiH_4 was varied, the rf power was maintained at 500 W and when the rf power was varied, the gas ratio of NH_3/SiH_4 was kept at 2. As shown in Fig. 2, when the gas ratio of NH_3/SiH_4 was increased from 2 to 14, the deposition rate decreased from 62 to 15 nm/min, due to the decrease of in the proportion of the silicon source in the gas mixture. Also, when the ICP source power was increased from 300 to 1 kW, the deposition rate increased from 27 to 70 nm/min, although the increase of the deposition rate appears to slowly saturate from 500 W. The slow saturation of the deposition rate with increasing ICP power appears to also be related to the depletion of the silicon source in the gas mixture at a high deposition rate (that is, silicon precursor limitation). In Fig. 2, the effect of the dc bias voltage in the range from 0 to -150 V on the deposition rate is also shown for different

gas mixture ratios of NH_3/SiH_4 . As shown in the figure, increasing the dc bias voltage to -150 V slightly decreased the deposition rate and, at -150 V, a decrease in the deposition rate of about 10% was observed for most of the gas mixtures. The decrease of the deposition rate with increasing dc bias voltage is believed to be related not only to the possible sputter etching during the deposition, but also to the increased density of the thin film deposited by the ion bombardment on the substrate.^{17,18)}

For the SiN_x thin films deposited using various gas mixtures at an ICP power of 500 W without a dc bias voltage, the chemical bonding states were investigated by FTIR and their FTIR broadband absorption spectra in the wavenumber range of $600\text{--}4000\text{ cm}^{-1}$ are shown in Fig. 3(a). The SiN_x thin films were deposited on p-type (100) silicon wafers and the thickness was maintained at 300 ± 20 nm. As shown in the figure, a strong Si–N band peak was observed at $880\text{--}900\text{ cm}^{-1}$,¹⁹⁾ and smaller peaks related to Si–O, Si–H, and N–H bonding were observed at 1170 , 2190 , and 3350 cm^{-1} , respectively.²⁰⁾ In addition, a small broad Si–O–Si bonding peak was also observed at about 1060 cm^{-1} on the shoulder of the Si–N peak. As shown in Fig. 3(a), increasing the gas mixture ratio of NH_3/SiH_4 decreased the bonding peak intensity related to Si–H, while increasing the peak intensity related to N–H bonding. The decrease of the Si–H bonding peak and increase of the N–H bonding with increasing NH_3/SiH_4 ratio are believed to be related to the bonding of the excess N in the gas mixture with H in the film. The hydrogen in the silicon nitride film is known to affect its dielectric characteristics. By forming Si–H instead of N–H, it can remove the dangling bonds in the film and improve the electrical properties, while the increase in the amount of N–H bonds increases the interface trap density and forms local energy levels in the band gap, which degrade the electrical properties of the deposited silicon nitride.²¹⁾ The oxygen related bonding peaks at 1060 and 1170 cm^{-1} , which may have originated from the background oxygen in the system, did not vary significantly with the gas mixture ratio. Increasing the gas mixture ratio of NH_3/SiH_4 caused the intensity of the Si–N bonding peak to be slightly increased, while shifting the peak position to the less silicon nitride-like bonding region.

Figure 3(b) shows the variation of the Si–N bonding peak position with the gas mixture ratio of NH_3/SiH_4 in Fig. 3(a) more clearly. In addition, the variation of the refractive index (n) of the deposited SiN thin film with the NH_3/SiH_4 ratio measured by an ellipsometer is also shown. As shown in Fig. 3(b), decreasing the NH_3/SiH_4 ratio from 14 to 2 decreased the peak wavenumber position from 907.5 to 883.3 cm^{-1} , bringing it closer to the more SiN-like bonding position at the expense of other bondings such as silicon oxide.²²⁾ The refractive index increased from 1.59 to 1.7 as the NH_3/SiH_4 ratio was decreased from 14 to 2, which is close to that of SiN. This also indicates the formation of more SiN-like bondings in the film. Therefore, by decreasing the NH_3/SiH_4 ratio to 2, a SiN-like thin film having a greater amount of Si–N bonding could be obtained.

In the case of an NH_3/SiH_4 ratio of 2 at an ICP power of 500 W, a dc bias voltage of up to -150 V was applied and the material characteristics were investigated. The FTIR absorption spectra for the Si–N bonding and the refractive

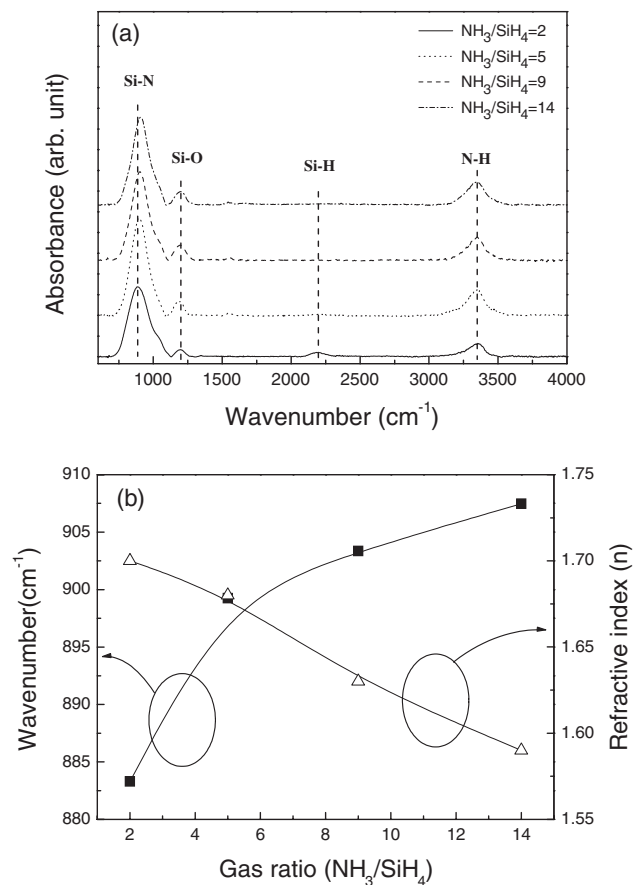


Fig. 3. (a) FTIR spectra of SiN_x films as a function of the NH_3/SiH_4 gas ratio for an rf ICP source power of 500 W and with no dc bias voltage. (b) Si–N band peak shift and refractive index of the SiN_x films as a function of the NH_3/SiH_4 gas ratio for an rf ICP source power of 500 W and with no dc bias voltage.

index of the deposited SiN_x measured as a function of the dc bias voltage are shown in Figs. 4(a) and 4(b), respectively. The thickness of the deposited SiN_x thin films was also maintained at 300 ± 20 nm. As shown in Fig. 4(a), as the dc bias voltage applied to the substrate was increased, the Si–N bonding peak position shifted to a lower wavenumber, varying from 883.3 cm^{-1} for no bias voltage to 864 cm^{-1} for a dc bias voltage of -150 V, although no significant shift of the wavenumber was observed at dc bias voltages higher than -100 V. In addition, the intensities of the Si–O bonding related peaks, such as those at 1060 cm^{-1} and 1170 cm^{-1} , decreased with increasing dc bias voltage. Therefore, it is believed that increasing the dc bias voltage during the deposition of the thin film increased the bonding ratio of Si–N/Si–O in the deposited film and made it more silicon nitride-rich.²³⁾ The refractive index measured as a function of the dc bias voltage in Fig. 4(b) increased from 1.7 at no bias voltage to 1.83 at a dc bias voltage of -150 V, which also indicates the formation of a silicon nitride-rich thin film. Kuo *et al.*¹⁸⁾ reported that the refractive index of the silicon nitride gate dielectric layer showing the lowest threshold voltage (V_{th}) of an MIS device is in the range of 1.8–1.9.

The Si/N ratio and the hydrogen concentration in the deposited film were estimated using an XPS and FTIR spectra in Fig. 3(a), respectively,^{24,25)} and the results are shown in Fig. 5. The Si/N ratio and the hydrogen concen-

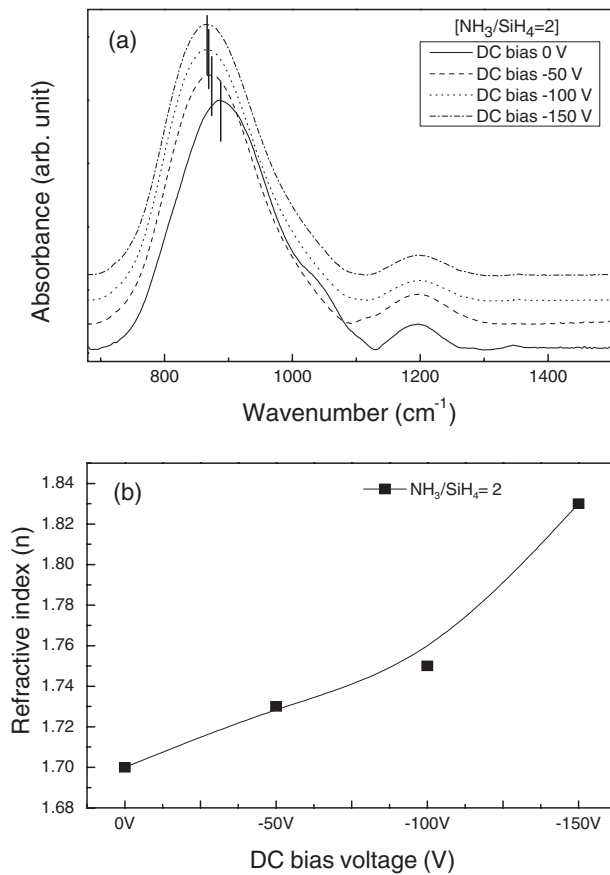


Fig. 4. (a) Si-N narrow band FTIR spectra of SiN_x films as a function of the dc bias voltage on the substrate for a gas ratio of NH₃/SiH₄ = 2 and rf ICP source power of 500 W. (b) Refractive index of the SiN_x films as a function of the dc bias voltage on the substrate for a gas ratio of NH₃/SiH₄ = 2 and rf ICP source power of 500 W.

tration in the film were obtained as a function of dc bias voltage for the condition of NH₃/SiH₄ = 2 and an ICP power of 500 W. As shown in Fig. 5, the total hydrogen concentration in the SiN_x thin film was mostly due to the N-H bonding compared to Si-H bonding, and the hydrogen concentration was decreased with the increase of dc bias voltage possibly due to the removal of hydrogen from the N-H bonding in the film by the ion bombardment. The decrease of hydrogen content in the SiN_x thin film is believed to increase the film density and is also related to the change of the film properties shown in Figs. 4(a) and 4(b) for the increase of Si-N bonding and the change of refractive index, respectively.²⁶⁾ In the case of the Si/N ratio in the film, as shown in the figure, it decreased with the increase of dc bias voltage, therefore, the film became more nitrogen-rich SiN_x thin film with the increase of dc bias voltage.

MIS capacitors composed of Al (100 nm)-SiN_x (300 nm)-p-type (100) silicon were fabricated with the SiN_x thin films deposited at various dc bias voltages, as shown in Fig. 4, and their high frequency (1 MHz) C-V characteristics were measured and the results shown in Fig. 6. As shown in Fig. 6, the C-V characteristics showed hysteresis under all dc bias voltage conditions, possibly due to the local distribution of the interface charge trap density at the interface between the silicon and SiN_x. The hysteresis is due to the differences in the gate voltages during the trapping and escaping of the carriers (electrons or holes) at the

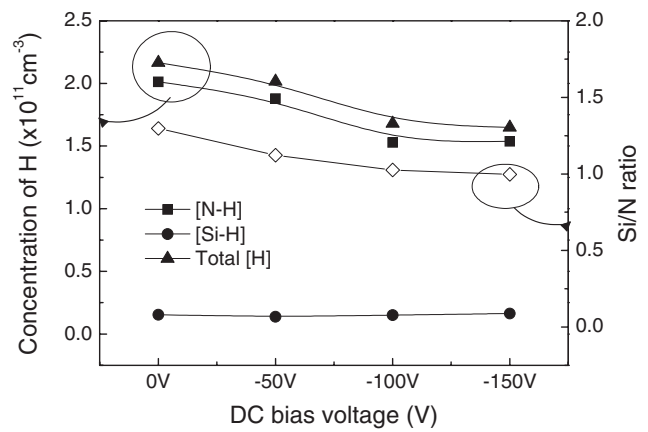


Fig. 5. Hydrogen concentration and Si/N ratio of the films as a function of the dc bias voltage for a gas ratio of NH₃/SiH₄ = 2 and an rf ICP source power of 500 W.

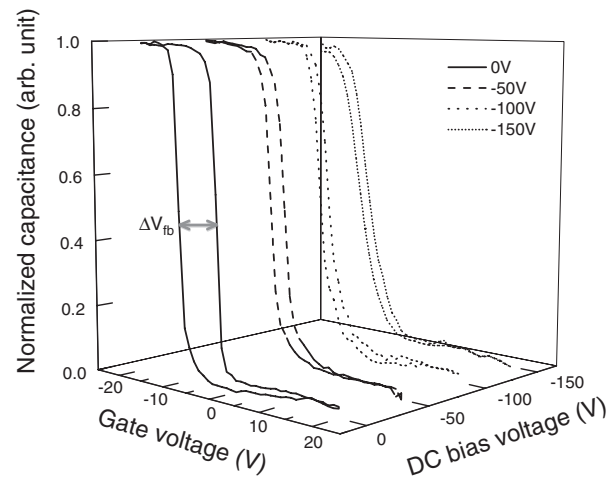


Fig. 6. High frequency (1 MHz) C-V characteristics of MIS structure with SiN_x films deposited at different dc bias voltages on the substrate. The voltage sweep is in the up-down direction from -20 to 20 V.

interface between SiN_x and silicon.²⁷⁾ Also, these are slow traps, because the response of the trapped carriers depends not only on the gate voltage, but also on the time of the voltage sweep.²⁸⁾ The amount of charge (Q) trapped in the slow interface trap level tends to shift the threshold voltage (V_{th}) of the TFT devices. As shown in Fig. 6, as the dc bias voltage was increased from 0 to -150 V, the shift of threshold voltage (ΔV_{fb}) caused by decreasing the hysteresis voltage was decreased from 8.1 to 1.8 V. Therefore, a lower interface trap charge density is believed to be obtained under higher dc bias voltage conditions.

Using the C-V characteristics shown in Fig. 6 and an ellipsometer, the interface trap charge density of the MIS capacitors and the dielectric constant of the deposited silicon nitride thin films were obtained as a function of the dc bias voltage for NH₃/SiH₄ = 2 and an ICP power of 500 W and the results are shown in Fig. 7. The trap charge density in Fig. 7 was obtained from the formula: $Q = \Delta V_{fb}(C_{di}/q)$, where ΔV_{fb} is the hysteresis voltage, C_{di} is the dielectric capacitance, and q is the elementary charge.²⁸⁾ When no dc bias voltage was applied, the trap density was as high as 1 × 10¹² cm⁻² and, by the application of a dc bias voltage

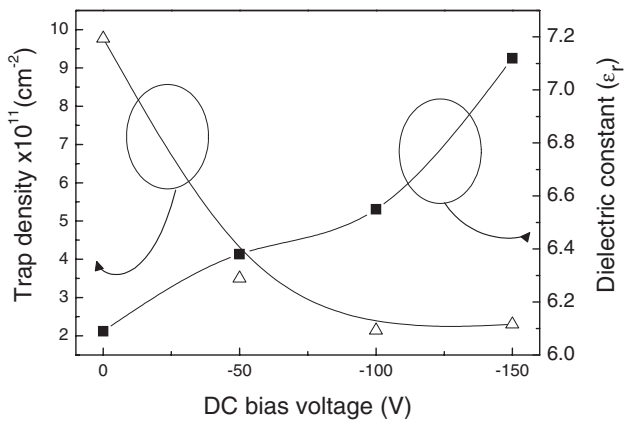


Fig. 7. The trap charge density at the interface between the SiN_x films and the bulk Si and the dielectric constant of the SiN_x films deposited at different dc bias voltages on the substrate.

of -100 V , the trap density was decreased to $2 \times 10^{11} \text{ cm}^{-2}$. Further increasing the dc bias voltage up to -150 V slightly increased the trap charge density. A high trap charge density degrades the electrical properties of the fabricated MIS devices by forming an internal electric field. The interface trap density is related to structural defects at the insulator/semiconductor surface and the density is expected to be higher for silicon-rich film and for less dense film. By the ion bombardment during the deposition of SiN_x film, the film density was increased and more nitrogen-rich SiN_x thin film was obtained. Therefore, the decrease of the interface trap density for the dc bias voltage lower than -100 V is believed to be related to the increase of film density and the increase of Si–N bonding at the interface. However, for the ion bombardment energy higher than -100 V , due to the increase of the film structural disorder by the high energy ion bombardment, the interface trap density was slightly increased.

4. Conclusions

Using an internal-type ICP source, SiN_x thin films were deposited at the temperature lower than 100°C at various NH_3/SiH_4 ratios and rf powers applied to the ICP source and the effects of these parameters on the film properties were investigated, in order to evaluate their potential use as the inorganic gate dielectric for organic devices and flexible display devices. In addition, to improve the characteristics of the deposited SiN_x thin film, a dc bias voltage was applied and its effect on the film properties was also investigated.

Increasing the rf power increased the SiN_x deposition rate, although the deposition rate at a fixed NH_3/SiH_4 ratio became nearly saturated at high rf powers, due to the limitation of the silicon source in the gas mixture. Increasing the NH_3/SiH_4 ratio at a fixed total gas flow rate also decreased the deposition rate, due to the limitation of the silicon source in the gas mixture. The SiN_x thin film deposited with a gas mixture ratio of $\text{NH}_3/\text{SiH}_4 = 2$ showed the lowest amount of N–H bonding and the largest amount of Si–N bonding. The further improvement of the properties of the deposited SiN_x film could be obtained by the application of a dc bias voltage of up to -150 V during the deposition process. Under deposition conditions of

$\text{NH}_3/\text{SiH}_4 = 2$ and a dc bias of -150 V , the refractive index was 1.83, which is close to that of silicon nitride (2.02). And, under these conditions, the interface trap charge density measured for the fabricated MIS capacitors was the lowest, with a value of $2 \times 10^{11} \text{ cm}^{-2}$ being obtained compared to the value of $1 \times 10^{12} \text{ cm}^{-2}$ in the case where no bias voltage was applied. Therefore, it is believed that high quality SiN_x thin film potentially applicable to flexible display devices can be fabricated at the temperature lower than 100°C using an internal-type NH_3/SiH_4 ICP with the application of a small dc bias voltage to the substrate.

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