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Low Hydrogen Content Silicon Nitride Films Deposited at Room Temperature with an ECR Plasma Source

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Silicon nitride layers with very low hydrogen content (less than 1 atomic percent) were deposited at near room temperature, from N_2 and SiH_4, with a multipolar electron cyclotron resonance plasma. The influences of pressure and nitrogen flow rate on physical and electrical properties were studied in order to minimize the hydrogen and oxygen content in the layers. The optimized layers were characterized by a refractive index of 1.98, a dielectric constant of 7.2, and Si/N ratio values of 0.78. The layers exhibited very good dielectric strength, which was confirmed by large breakdown fields of 12 MV/cm, very high resistivities of 10^{16} Ω cm, and maximum charges to breakdown values of 90 C/cm^2. Increasing the deposition pressure and decreasing the N_2 flow improved the SiN/Si interface, due to increased oxygen incorporation. The dominant conduction mechanism in the layers was the Poole-Frenkel effect. The critical field and the trap energy had similar dependencies on deposition pressure. Fowler-Nordheim tunneling occurred at high gate biases, for the layers deposited at the highest pressure of about 22 mTorr.
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Decreasing the temperature at which dielectrics are deposited without causing any deterioration of the dielectrics properties has become a priority in the thin film research area. Low-temperature deposition is needed, for example, for producing thin film transistors (TFTs) on ultrathin, plastic substrates that can withstand temperatures of up to 100°C.[1,2] Such flexible substrates may replace the glass screens in future displays due to several advantages, *e.g.*, lower cost, lower power consumption, lower weight, and better flexibility.

The standard gate dielectric nowadays for amorphous TFTs is silicon nitride (Si_3N_4) deposited by radio-frequency plasma enhanced chemical vapor deposition (rf-PECVD). However, the processing temperature of rf-PECVD is 300°C, which is too high for flexible displays. Furthermore, these layers exhibit high hydrogen contents of up to 20 atomic percent (atom %) and low dielectric strength. In contrast, electron cyclotron resonance (ECR) PECVD Si_3N_4 layers with good material and electrical properties have been successfully deposited in recent years, at much lower temperatures.[3-5] ECR plasma operates at a low pressure, has a low electron and ion energy, and a high degree of ionization.[6,7] Because of these soft and dense characteristics of the ECR plasma,[8] the deposition temperature can be lowered, while reducing the concentration of unwanted hydrogen bonds through ion bombardment.

Although very good quality Si_3N_4 films with good interfaces and high breakdown fields have been deposited at room temperature using divergent ECR plasma sources and distributed ECR plasma sources,[3,9,10] the level of hydrogen contamination could not be reduced below 5 atom %.

A less known ECR discharge configuration, called multipolar ECR[11] has been shown to reduce the hydrogen content even more. This new technique has certain advantages such as a magnetic field parallel to the substrate, which minimizes the plasma damage, a tunable microwave cavity, and a quartz dome that reduces the metal contamination. To the best of our knowledge, only one paper dealing with the current-voltage (I-V) characteristics of Si_3N_4-deposited layers with a multipolar ECR plasma source has been published.[12] The influences of different deposition parameters upon the electrical properties of Al/SiN/Si structures were not studied in detail.

Several studies have shown the presence of oxygen contamination in ECR nitrides[3,10,12-17] and the attempts to minimize the oxygen content, by reducing the sputtering process at low microwave power.[17] However, the reduction of the oxygen contamination, by modifying the deposition pressure and improving the dissociation of nitrogen, was not investigated in detail.

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Our aim is to obtain layers with less than 1 atom % hydrogen contamination at room temperature by multipolar ECR PECVD, without any annealing process. Helium is known to improve the film properties and efficiently eliminate hydrogen in rf-PECVD layers.[18] Hence 2% silane diluted in helium was employed as a gas precursor in our deposition system.

In this paper, the influences of the total pressure and the nitrogen flow on deposition kinetics, film composition, and capacitance-voltage (C-V) and I-V measurements were investigated in order to find the optimal deposition conditions. We focused on the reliability, the conduction mechanism, and the C-V characteristics of ECR PECVD Si_3N_4, because there is not much literature available on these aspects. Correlations between the total pressure, the oxygen content in the layers, and the electrical behavior of the films were studied.

Experimental

The deposition system consists of a microwave plasma disk reactor (MPDR-300), comparable with the one described by Asmussen.[11] A multipolar or multicusp plasma is created by a microwave source of 2.45 GHz and eight permanent magnets with alternating polarity surrounding the chamber. Since the magnetic field is parallel to the substrate, the wafer situated downstream of the plasma is not placed in a diverging magnetic field. Consequently, the ions and electrons bombardment is greatly reduced in the multipolar ECR plasma system. The base pressure in the reaction chamber was $3 \cdot 10^{-6}$ Torr.

Electronic pure nitrogen (N_2) and 2% silane (SiH_4) diluted in helium were used as gas precursors. N_2 was admitted directly into the quartz dome, while SiH_4 was introduced through a dispersive ring downstream from the plasma. The silicon nitride layers were obtained at near room temperature, without external heating. The substrate temperature was measured with a thermocouple bonded inside the sample holder. Due to plasma heating, the temperature reached a maximum of 60°C. During the experiments, the flow rate of 2% SiH_4 in He was maintained at a constant value of 5 standard cubic centimeters per minute (sccm) and the microwave power was set at 400 W. The total pressure was in the range of 5-22 mTorr, and the flow rate of nitrogen varied between 30 and 110 sccm. Silicon nitride films with thicknesses of 30-50 nm were deposited on 3 in. (100)-oriented, n-type silicon (Si) wafers, with a resistivity of 1-10 Ω cm. The wafer preparation included a 10 min dip in 100% fuming HNO_3 and a 10 min dip in boiling 69% HNO_3, followed by a dip in 1% HF, in order to remove the native oxide.

The thickness and the refractive index of the deposited layers were determined using an automatic Plasmos ellipsometer, with a fixed wavelength of 633 nm. A PHI Quantum 2000 scanning elec-

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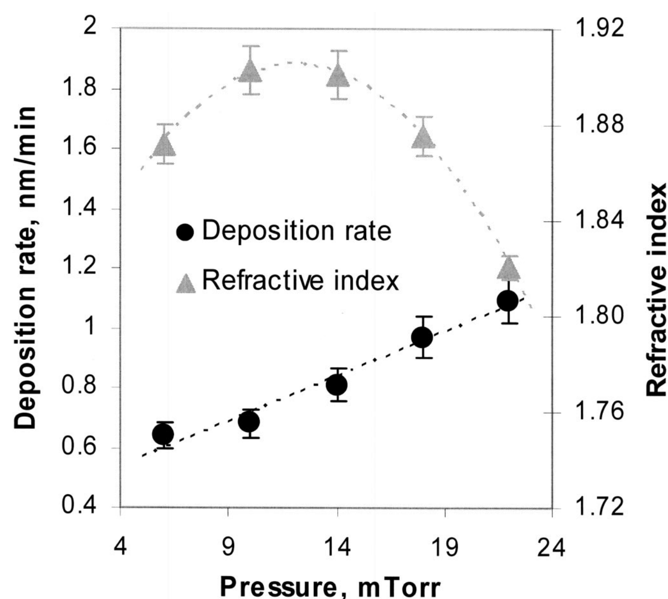

Figure 1. Deposition rate and refractive index *vs.* total pressure for layers deposited with 5 sccm SiH_4/He, 30 sccm N_2, and 400 W.

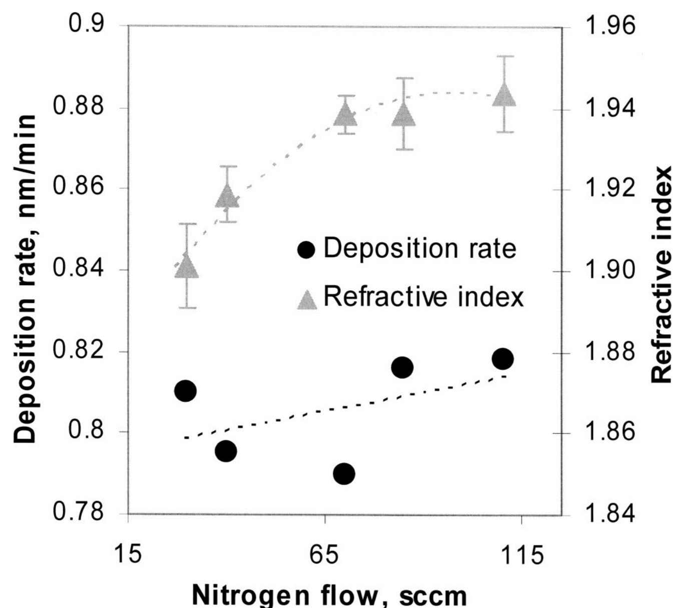

Figure 2. Deposition rate and refractive index *vs.* nitrogen flow for layers deposited with 5 sccm SiH_4/He, at 14 mTorr, and 400 W.

tron spectroscopy for chemical analysis (ESCA) microprobe was used to determine the chemical composition with an error of 3% by X-ray photoelectron spectroscopy (XPS). The hydrogen content in the films was analyzed by elastic recoil detection (ERD).

Metal-insulator-silicon (MIS) capacitors were manufactured by sputtering an aluminum layer of 1 μm, followed by lithography and etching processes. An aluminum layer of 1 μm was also sputtered on the back side of the Si wafer, after removing the native oxide with buffered oxide etchant (BOE), in order to form a better contact. All samples were subjected to an aluminum sintering step of 5 min, at 400°C in wet N_2 ambient (N_2 bubbled through DI water at room temperature) in order to ensure a good ohmic contact. The high frequency (10 kHz) and quasistatic *C-V* measurements were performed with a Hewlett-Packard 4275A multifrequency meter and a Hewlett-Packard 4140B pA meter, respectively. A Hewlett-Packard 4156 parameter analyzer was used for the *I-V* and the constant current stress (CCS) measurements. In both cases, a positive voltage was applied to the gate, thus electrons were injected from the substrate. The critical and breakdown fields were extracted by averaging the *I-V* curves of 25 capacitors of 0.1 mm^2, with a statistical error of 3%. Weibull plots were drawn from 50 CCS measurements, performed on 0.01 mm^2 capacitors by forcing a constant current of 1 mA/cm^2 through the dielectric.

Results and Discussion

Several papers have shown that it is difficult to obtain layers at room temperature with concomitantly good stoichiometry, good interface trap densities, low trapped charge densities, and high breakdown fields.[8,9] In order to obtain an optimized film, we studied the influence of the total pressure in the chamber and the nitrogen flow on several of the Si_3N_4 properties like deposition rate, refractive index, film composition, and interface and bulk electrical properties.

Deposition kinetics.—It can be seen from Fig. 1 that the deposition rate increases linearly with the pressure, for total pressures between 5 and 22 mTorr. During this experiment, the partial pressures of the gases increased (for SiH_4 from 0.014 to 0.062 mTorr, for He from 0.7 to 3.08 mTorr, and for N_2 from 4.28 to 18.85 mTorr). Thus, the concentration of the molecules and radicals inside the chamber also increased.[19] The proportionality relation between the deposition rate and the concentration of chemical radicals can therefore explain the trend observed in Fig. 1.[12,20]

The nitrogen flow rate has no significant influence on the deposition rate (Fig. 2), despite the fact that the silane partial pressure becomes nearly three times smaller (from 0.04 to 0.012 mTorr) at high N_2 flows. The nitrogen partial pressure increases only from 12 to 13.4 mTorr, while the total pressure is kept constant at 14 mTorr. The data apparently suggests that the deposition rate is more or less independent on the silane partial pressure for these deposition conditions (microwave power of 400 W, very low silane partial pressure). This result is in disagreement with other works reported in the literature[5] and our prior results. While, for our previous films, deposited at a lower microwave power of 150 W, a decrease in deposition rate from 0.87 to 0.59 nm/min with increasing the nitrogen flow rate from 10 till 75 sccm was observed, in this case apparently the concentration of SiH_x radicals at the surface remains unchanged. It is possible that more SiH_4 molecules are excited at higher N_2 flow and higher microwave power, due to a higher concentration of nitrogen radicals arriving from the plasma, thus maintaining the deposition rate constant.

Deposition rates of 1 nm/min as recorded in our experimental setup are suitable for the deposition of very thin gate dielectric layers for TFTs, with reproducible properties.

Film composition.—The refractive index decreases from 1.9 to 1.8 with increasing the total pressure (Fig. 1) probably due to modifications in film composition (oxygen and hydrogen contamination or excess nitrogen). The XPS measurements indicate that more oxygen and less nitrogen are incorporated in the deposited films at high pressure (Fig. 3). Several studies have indicated the presence of oxygen (1-20 atom %) in silicon nitride layers deposited with an ECR plasma,[3,10,12-17] and the cause was found to be either the sputtering of the quartz dome,[3,17] insufficient vacuum conditions,[12,13] or oxidation of highly porous films after deposition, in ambient.[14,16]

In our case, the increase of oxygen content with pressure can be explained either by a higher porosity at higher pressure due to less ion bombardment,[21] or by a higher degree of deexcitation of nitrogen radicals at high pressure.[12] The first hypothesis was not confirmed by composition measurements done at various periods of time after deposition. Also the lack of hydrogen atoms observed in ERD measurements (Table I) indicates that the layers are very dense, thus the incorporation of oxygen took place during deposition.

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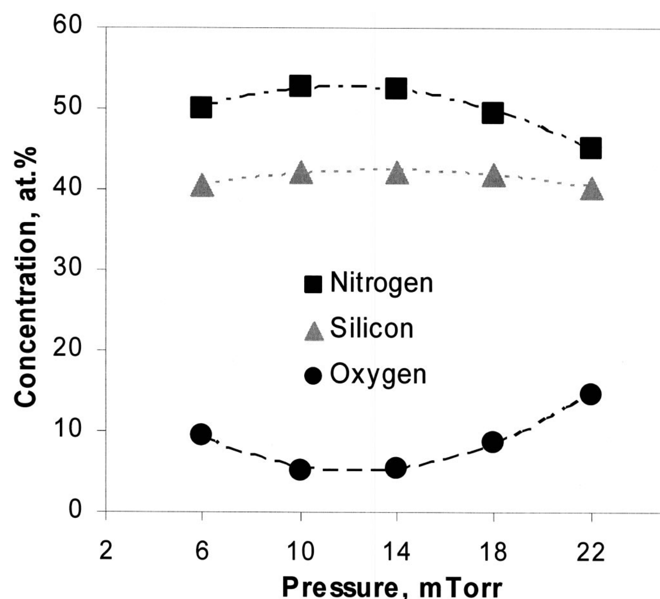

Figure 3. Atomic composition *vs.* pressure for layers deposited with 5 sccm SiH_4/He, 30 sccm N_2, and 400 W.

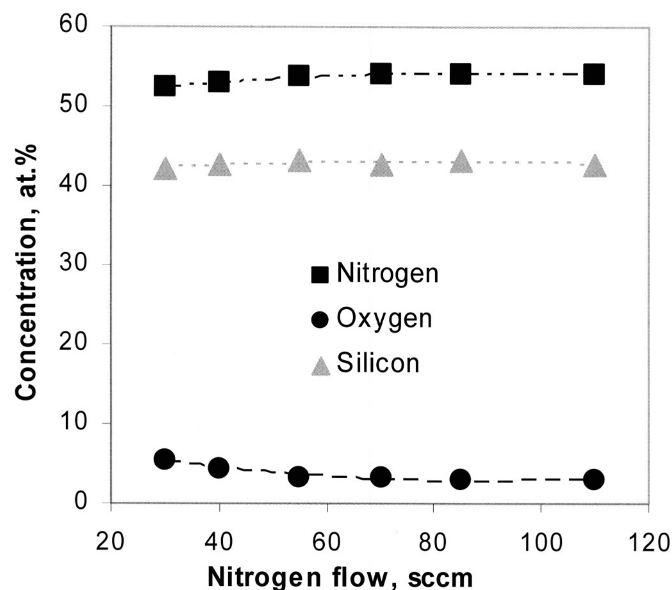

Figure 4. Atomic composition *vs.* nitrogen flow for layers deposited with 5 sccm SiH_4/He, at 14 mTorr, and 400 W.

Previous experiments have shown that high microwave powers of 400 W and low pressures may also cause sputtering of the quartz dome. This explains why the film deposited at the lowest pressure of 6 mTorr contains more oxygen than the layer obtained at 10 mTorr. Decreasing the microwave power below 200 W[17] and improving the vacuum[13] should prevent any oxygen contamination.

Higher nitrogen flow caused an elevated concentration of nitrogen radicals in the plasma. Thus, more nitrogen was incorporated in the layers and the refractive index increased to 1.95 (Fig. 2 and 4). It is difficult to measure accurately, with a constant wavelength, the refractive index of such thin layers. Therefore, a thicker layer of 85 nm was grown, at the same deposition conditions, and the measured refractive index increased from 1.95 to 1.98. This is a value comparable to the refractive index of the silicon nitride obtained at 800°C by low pressure chemical vapor deposition (LPCVD).[22]

The oxygen contamination was reduced to 2 atom % for a total pressure of 14 mTorr and a nitrogen flow rate of more than 55 sccm (Fig. 4).

Both Si-H and N-H absorption peaks in the infrared spectra were too weak to determine the hydrogen concentration; therefore the hydrogen content was measured by ERD (Table I). The total hydrogen contamination was less than 1 atom %. The silicon nitride layers were deposited at room temperature and did not experience any annealing process before the composition measurements. Nevertheless they exhibited the lowest hydrogen content, lower that could be found in literature, proving that multipolar ECR PECVD is a very good method for obtaining films with a low thermal budget and good properties. For comparison, the hydrogen content of a 800°C LPCVD silicon nitride layer, measured with the same ERD equipment, was 3.3 atom %.[22] The source of hydrogen in the deposited films is SiH_4. SiN_xH_y radicals are adsorbed at the film surface, and they migrate on the surface until they encounter nitrogen radicals, needed for formation of Si_3N_4 and elimination of hydrogen. At low temperatures, however, there is not sufficient energy for the particles to migrate, therefore the hydrogen atoms are not entirely eliminated, and the final product is not stoichiometric. In case of ECR PECVD layers, due to a high density plasma and a better energy transfer from the plasma toward the substrate,[8] there is an improvement in the hydrogen desorption process.

Table I. Atomic composition of silicon nitrides deposited with 5 sccm SiH_4/He and 400 W.

Conditions	Silicon (atom %)	Nitrogen (atom %)	Oxygen (atom %)	Hydrogen (atom %)	Si/N ratio
6 mTorr, 30 sccm N_2	39.9	49.2	10	0.9	0.81
14 mTorr, 30 sccm N_2	43.4	50.1	5.5	1.0	0.87
14 mTorr, 40 sccm N_2	41.6	53.1	4.3	1.0	0.78
14 mTorr, 110 sccm N_2	44	52.3	3.1	0.6	0.84

C-V characteristics.—The positive effective charge calculated from the flatband voltage and the interface trap density determined by the Castagne method[23] are plotted *vs.* total pressure and nitrogen flow rate in Fig. 5 and 6, respectively. As it can be seen, the influences of the deposition parameters upon the interface properties, the trapped charge, the refractive index, and the oxygen content are all very well correlated (Fig. 1-6). Since silicon nitride has a more rigid structure than silicon oxide, it is also characterized by a higher concentration of silicon dangling bonds and a more strained interface with silicon. An increase in the oxygen concentration, as observed for the layers deposited at high pressure, can relax the stressed interface, and decrease the effective charge and the interface trap density (Fig. 5).

Compared to LPCVD Si_3N_4 layers deposited at 800°C and other layers obtained by ECR at room temperature,[9] our dielectrics have lower effective charge. However, the interface trap density is higher, probably due to the absence of hydrogen, which is known to saturate the dangling bonds present at the interface.[9] The hysteresis shown in the high-frequency C-V measurements was only 0.01 MV/cm for the layers obtained at high pressure, and 0.085 MV/cm for the films obtained at high pressure and high nitrogen flow. These values are much lower than the ones calculated for rf-PECVD nitrides or ECR nitrides.[24]

I-V characteristics.—The influence of total pressure upon the electrical strength of the layers is presented in Fig. 7. The thickness of the layers was around 42 nm. The critical field is the electric field for which the current that passes through the capacitor is higher than 10^{-8} A/cm^2. The breakdown field is calculated as the electric field for which the current is 10 A/cm^2. The maximum breakdown field

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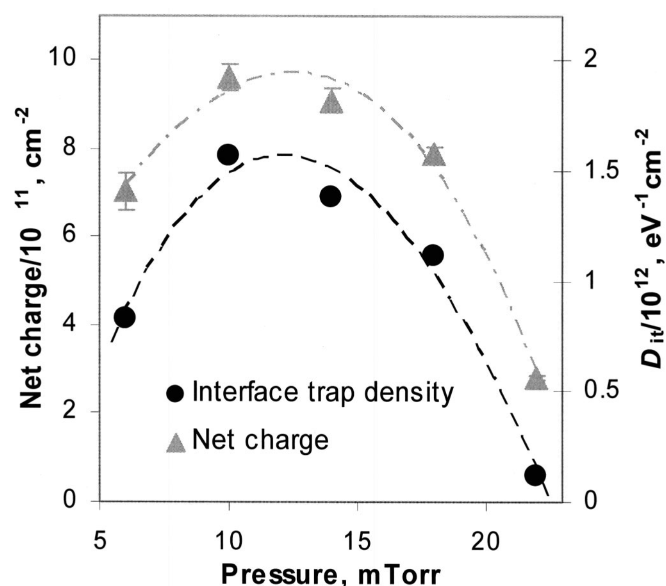

Figure 5. Dielectric constant and net charge *vs.* pressure for layers deposited with 5 sccm SiH_4/He, 30 sccm N_2, and 400 W.

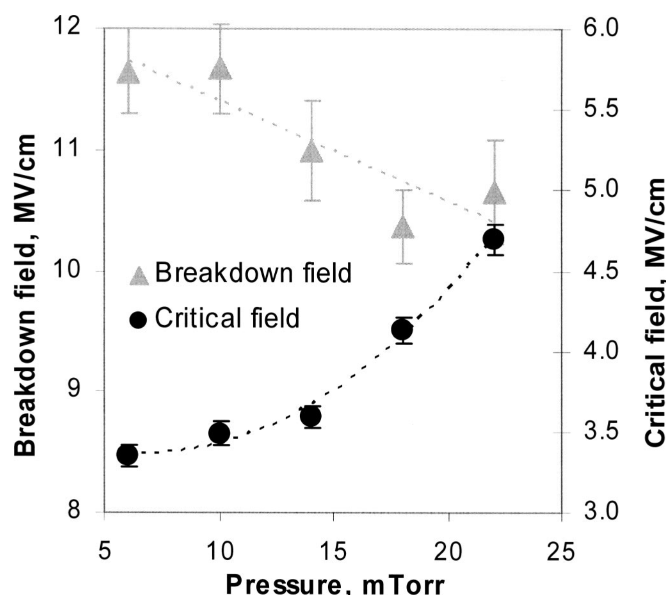

Figure 7. Breakdown and critical field *vs.* pressure for layers deposited with 5 sccm SiH_4/He, 30 sccm N_2, and 400 W.

was reached at around 12 MV/cm, which is much higher than the breakdown fields for the 300°C rf-PECVD layers[14] and for other Si_3N_4 layers obtained by ECR.[9,15] This is probably due to the low hydrogen contamination present in our films. It is well-known that the ion energy, which is responsible for densifying the layer and creating stronger bonds between the atoms, decreases with increasing the total pressure due to more collisions.[3] Probably due to a lower efficiency in energy transfer from the plasma toward the substrate at high total pressure,[3,8,12] the films exhibit lower breakdown fields.

The critical field increases for high deposition pressure (Fig. 7), most likely due to higher oxygen concentration. Si_3N_4 films exhibit critical fields of 4 MV/cm,[24] while SiO_2 has a higher critical field of around 6.5 MV/cm[25] due to different conduction mechanisms. Thus, the oxynitrides obtained at higher pressure exhibit larger critical fields, due to higher oxygen content (Fig. 7).

The current passing through the nitride can be represented by a straight line in the Poole-Frenkel graph [$\ln(J/E)$ *vs.* $E^{1/2}$], where J is the current density and E the electric field. Therefore, in Fig. 8, we have fitted the measured current with a Poole-Frenkel (PF) current.[26]

$$J_{PF}(E) = J_0 E \exp\left(\frac{-q\phi_T}{kT}\right)\exp\left[\frac{E^{1/2}}{rkT}\sqrt{\frac{q^3}{\pi\varepsilon_0\varepsilon_R}}\right] \quad [1]$$

where q is the electron charge, k the Boltzmann's constant, T the temperature, ϕ_T denotes the barrier height of the trap, $\varepsilon_0\varepsilon_R$ is the dielectric permittivity of Si_3N_4, and r and J_0 are constants. In order

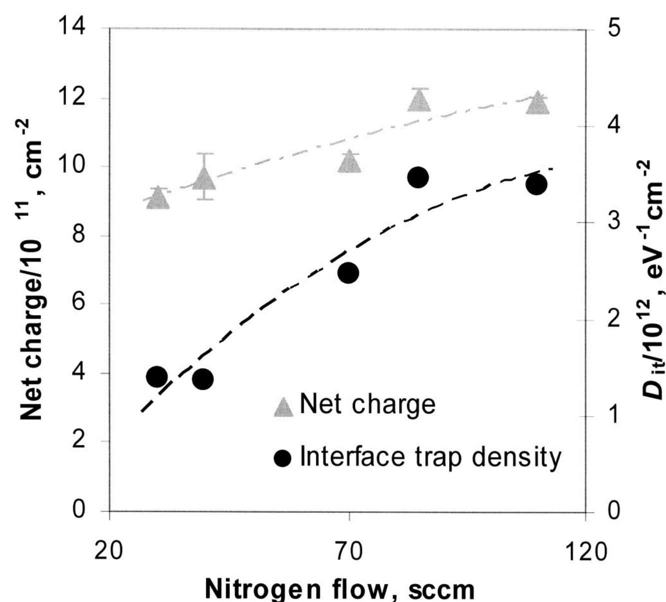

Figure 6. Net charge and interface trap density *vs.* nitrogen flow for layers deposited with 5 sccm SiH_4/He, at 14 mTorr, and 400 W.

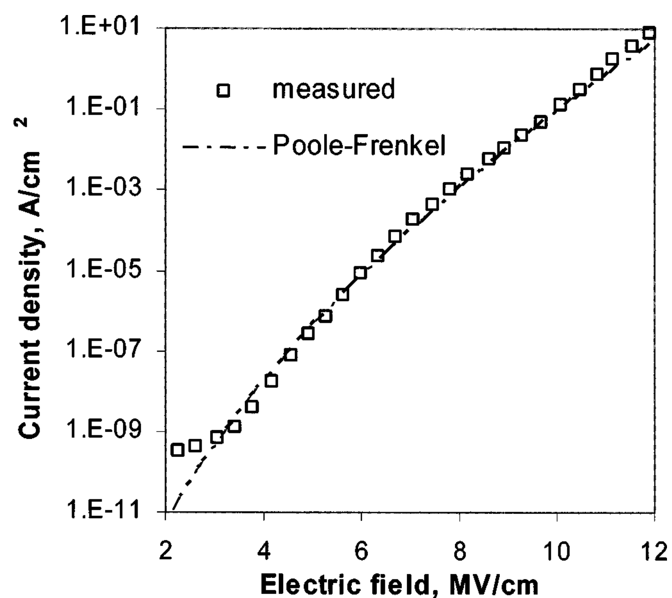

Figure 8. Measured current density *vs.* electric field for a layer deposited with 5 sccm SiH_4/He and 30 sccm N_2, at 14 mTorr, and 400 W; and the Poole-Frenkel current fitted, $\phi_T = 0.7$ V and $r = 1.3$.

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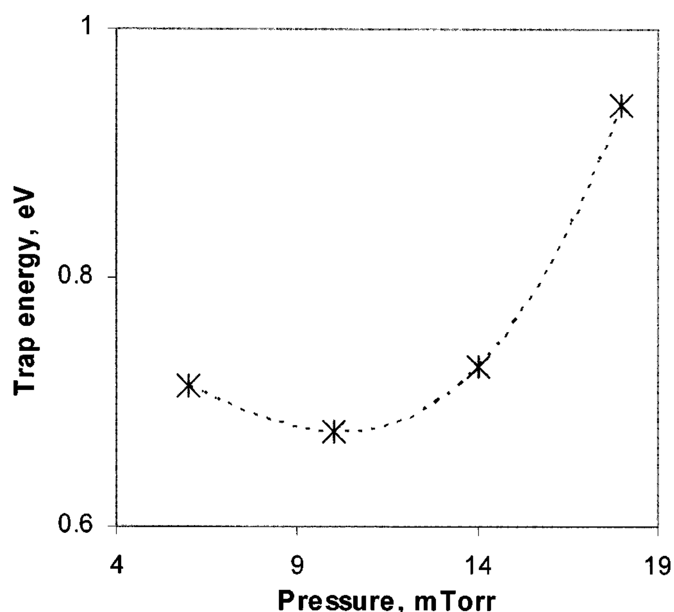

Figure 9. The trap energy extracted from PF fitting of *I-V* characteristics for layers deposited with 5 sccm SiH_4/He, 30 sccm N_2, and 400 W.

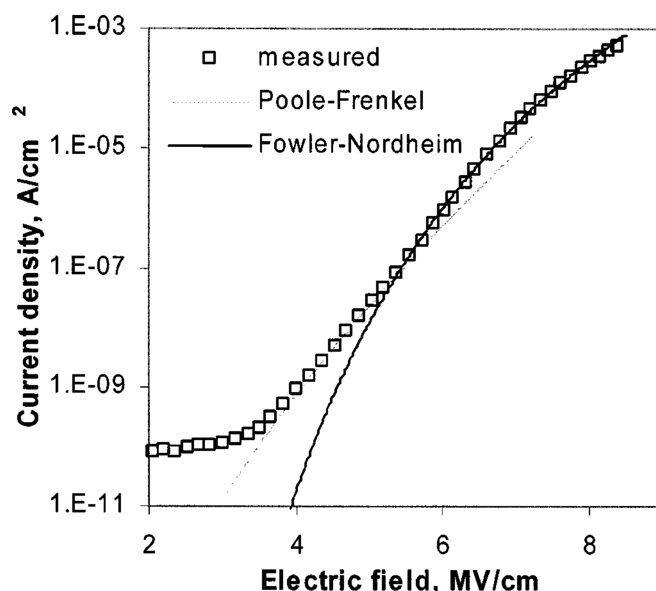

Figure 10. Measured current density *vs.* electric field for a layer deposited with 5 sccm SiH_4/He and 30 sccm N_2, at 22 mTorr, and 400 W; and the fitted Poole-Frenkel and Fowler-Nordheim currents, $\phi_T = 1$ V and $\phi = 2$ V, respectively.

to verify the conduction mechanism, the current was measured for four substrate temperatures (22, 62, 102, and 142°C), and an almost perfect fitting was observed for each temperature with: $\phi_T = 0.7$ V, $\varepsilon_R = 7$, and $r = 1.3$.

The trap energy $q\phi_T$, extracted from the *J-E* curves, increases from 0.67 to 0.94 eV with increasing the total pressure, as it can be seen in Fig. 9. This suggests that in case of higher oxygen incorporation, the traps are situated deeper into the bandgap and the probability of PF emission decreases. Consequently, the critical field, which indicates the electric field for which the current starts to increase, will be larger for layers obtained at higher pressure (Fig. 7). The current passing through the nitride deposited at the highest pressure of 22 mTorr, containing 15 atom % O could not be fitted at high biases with the Poole-Frenkel (PF) current. Instead, the current measured at high electric fields, was fitted with a Fowler-Nordheim (FN) tunneling current, for a barrier energy of 2 eV (Fig. 10). At high biases, the probability of FN tunneling appears to be larger than the probability of emission from the deep traps situated in the dielectric bandgap.

To the best of our knowledge, the reliability of silicon nitride layers deposited by multipolar ECR PECVD was not studied and nor presented until now. In Fig. 11, the charge-to-breakdown distributions (Weibull plots) for two silicon nitride films, deposited with different nitrogen flow, show a maximum charge-to-breakdown value of 90 C/cm^2. It can be seen that the layer deposited with lower N_2 flow shows improved reliability, contrary to the fact that nitrides possess higher charge-to-breakdown than oxides. It is possible that a higher energy transfer from the plasma at low flows is responsible for this effect. Substantiating this hypothesis requires more research.

Conclusions

High quality Si_3N_4 films with extremely low hydrogen content and very good dielectric strength were deposited at near room temperature. The layers deposited at rates of 1 nm/min with a multipolar ECR plasma source possess a refractive index of 1.98, a dielectric constant of 7.2, 2 atom % oxygen content, and 0.6 atom % hydrogen content. In order to optimize the deposition process, the effects of pressure and nitrogen flow on film properties were studied. The hydrogen content in our layers was lower than those reported in the literature for other Si_3N_4 layers. The oxygen content was minimized at low pressure and high N_2/SiH_4 ratios, due to an increased efficiency in nitrogen dissociation. The layers exhibited good electrical properties such as a breakdown field of 12 MV/cm, a resistivity of 10^{16} Ω cm, a charge to breakdown of 90 C/cm^2, and a net charge density less than 10^{12} cm^{-2}. The extremely low hydrogen contamination was responsible for a very good electric strength and film reliability, but also for an inferior interface. The effective charge, the interface trap density, and the trap energy were very well correlated with the oxygen concentration. The main conduction mechanism is Poole-Frenkel. For layers obtained at high pressure, with high oxygen contamination, Fowler-Nordheim tunneling appears at high biases, due to higher trap energy. The high-quality silicon nitride films obtained at near room temperature with ECR PECVD makes this

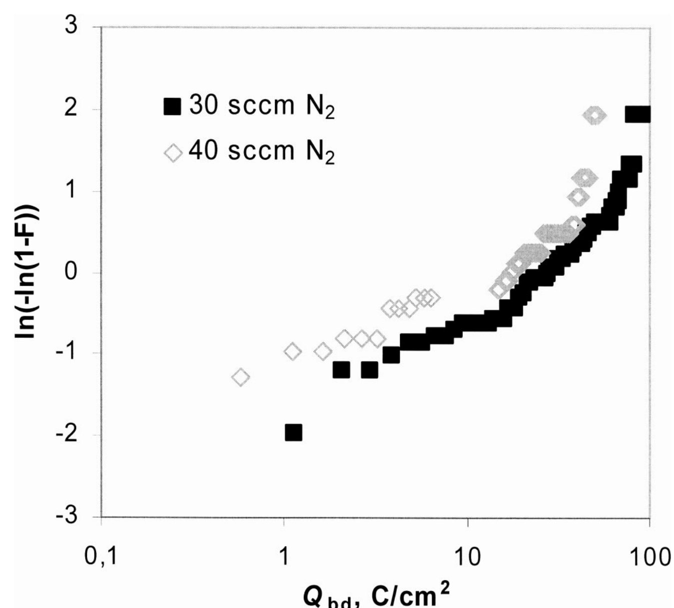

Figure 11. Charge-to-breakdown distributions (Weibull plot) for layers deposited with 5 sccm SiH_4/He, at 14 mTorr, 400 W, and different nitrogen flow.

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technology a good candidate for deposition of gate dielectrics and passivation layers for applications where low temperature processing is required.

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