Optimization of Ar-diluted N2 electoron cyclotron resonance plasma for high-quality SiN film growth at low temperature

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Optimization of Ar-diluted N₂ electron cyclotron resonance plasma for high-quality SiN film growth at low temperature

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The growth characteristics and electrical properties of a thin amorphous SiN film formed by Ar-diluted N₂ electron cyclotron resonance. (ECR) plasma irradiation at a low temperature of 400°C have been investigated. It is found that SiN film quality is sensitive to the N₂ mixing ratio [N₂/(N₂+Ar)] and a film having a structure nearest to stoichiometric can be obtained with 60% N₂ mixing plasma. An optimized substrate bias contributes to suitable plasma etching effect, which leads to the lowest leakage current density of SiN film with a given equivalent oxide thickness (EOT). Two kinds of impurities, N₂ molecules and Ar atoms, are found in the as-grown SiN film. The N₂ molecules concentration in SiN film has decisive influence on the film quality. An atomically flat interface between Si and SiN film are shown by high-resolution transmission electron microscope micrograph. The as-grown SiN film shows a leakage current more than two orders of magnitude lower than that of thermally grown SiO₂ having the same EOT.

Key words: *Si Nitride, Electron Cyclotron Resonance Plasma, Film Growth, Gate Dielectric*

1. Introduction

Si dioxide (SiO₂) films are widely used in the fabrication of ultra large scale integrated (ULSI) circuits. The advantages of SiO₂ include the high receptivity, excellent dielectric strength, a large band gap, and a high melting point. In addition, the Si/SiO₂ interface, which forms the heart of the Si-based metal-oxide-semiconductor field effect transistor (MOSFET) gate structure, is the most technologically important. However, with the scaling of the MOSFET for low power devices, SiO₂ gate dielectrics having thickness of less than 2 nm is required. When the physical thickness of SiO₂ gate approaches less than 2 nm, the leakage current increases because of direct tunneling phenomenon, which is the big obstacle for low power devices and also leads to poor device reliability.[1, 2]

In order to get thicker gate dielectrics and maintain high gate capacitance, high permittivity (high-*k*) materials have been strongly

required for alternative gate dielectrics to maintain the shrinking of ULSI.[2] Many researches have been done for high-*k* materials to replace the SiO₂ gate dielectric film and many candidates such as HfO₂,[3, 4] ZrO₂,[5, 6] TiO₂,[7] Ta₂O₅,[8] Al₂O₃,[9] La₂O₃,[10] Y₂O₃,[11] and Si₃N₄[12-14] have been proposed. In particular, ZrO₂ and HfO₂ seem to be the most attractive ones among those candidates.[2] However, there are still many problems in the application of these high-*k* dielectrics, in particular, their low immunity for oxygen and boron diffusion. Most of the high-*k* oxides are thermally unstable so that thin layers of SiOₓ are formed when they are directly contacted with Si substrate.[15-18] Furthermore, for *p*-channel MOSFET, boron penetration from the gate electrode into the Si substrate through the gate dielectrics causes threshold voltage shift and dielectric degradation.[19]

We believe that utilization of very thin SiN film is essentially important for high-*k* gate dielectrics, because SiN film has good properties such as higher permittivity and better barrier for oxygen and boron diffusion. Thus, SiN film would be a reasonable replacement of SiO₂ film as gate dielectric, if the interface quality can be improved. In addition, SiN film

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also can be used as a good interlayer between Si substrate and high-k material to improve the resistance of the high-k gate dielectric film for oxygen and boron diffusion in a gate stack structure.[20]

Recently, we have successfully fabricated the high-quality SiN films having the physical thickness of 3.9 nm by using Ar/N_2 mixed electron cyclotron resonance (ECR) plasma irradiation[21] at a low temperature of 400°C, which shows the effective oxide thickness (EOT) of 2.5 nm and a leakage current more than two orders of magnitude lower than that of thermally grown SiO_2 having the same EOT. However, the effects of N_2 mixing ratio and substrate bias of Ar-diluted N_2 ECR plasma on SiN film growth are not reported yet.

This paper is organized as follows: In Sec. 2, we present details of the experimental procedures. In Sec. 3, first, we show that the fabricated film is nearly stoichiometric SiN film; second, we show the effect of N_2 mixing ratio on growth characteristics and discuss on the influence of N_2 and Ar impurities in the film; third, we show the effect of substrate bias on growth characteristics and discuss the role of the substrate bias; fourth, we show the electrical properties and transmission electron microscopy (TEM) observation results for the SiN films fabricated under the optimum conditions and discuss the mechanism for the atomically flat interface.

2. Experimental

An ECR plasma irradiation apparatus is schematically shown in Fig. 1. A microwave power of 500 W at a frequency of 2.45 GHz was introduced into the plasma chamber via two E-plane Y-divider rectangular waveguides. The plasma chamber was surrounded by two ad-
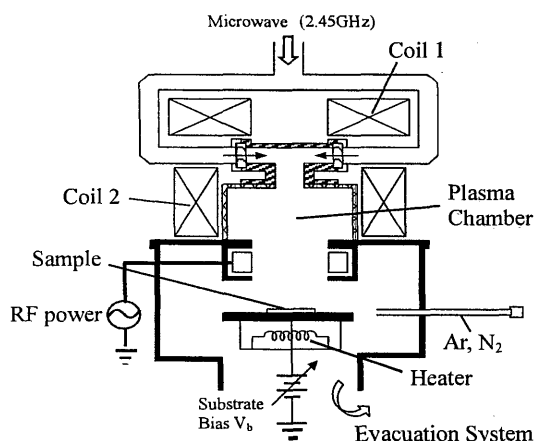

Fig. 1 A schematic diagram of the ECR plasma system.

justable magnetic coils, which generated a static magnetic field strength of 875 G in the chamber center. This magnetic field strength was chosen to achieve the ECR condition matched with the right-hand polarized microwave field. The substrate temperature was monitored by a thermocouple attached to the rear of the sample holder and well controlled as low as 400°C during the irradiation by adjusting the holder heating power. The plasma processing gases, Ar and N_2, were introduced into the system through the deposition chamber connected to the plasma chamber. The flow rates were controlled separately to change the N_2 mixing ratio $[N_2/(N_2+Ar)]$ from 5 to 100%. The total gas working pressure in the deposition chamber was fixed at 0.12 Pa. A DC-bias power (V_b) was supplied to the substrate for independent control of ion energies. This bias technique enables us to actively control the ion energies, leading to control of the film properties.

The substrate used in this study was n-type Czochralski grown single crystal Si with (100) orientation, having resistivity in the range of 8-12 Ω·cm and thickness of 650 μm. After the Si substrate was cleaned by standard RCA cleaning process, the Si substrate was dipped in a HF solution and rinsed in de-ionized water to remove the natural oxide before loading into the deposition chamber.

The electrical properties of SiN films were characterized by MOS capacitors with an evaporated Al electrode having an area of 2.25×10^{-4} cm^2 fabricated by lithography. The EOT was obtained from accumulation capacitance by high frequency (10 kHz) capacitance-voltage $(C\text{-}V)$ measurement. Current density-gate voltage $(J\text{-}V)$ characteristics were measured using a ramp positive voltage on the Al gate electrode, which corresponded to electron injection from Si substrate.

To investigate the structure of the SiN film, the refractive index and physical thickness of the SiN film were measured by ex situ spectroscopic ellipsometry (SE). To observe the flatness of the Si/SiN interface, high-resolution TEM (HRTEM) measurement was carried out. In addition, X-ray photoelectron spectroscopy (XPS) measurements were also performed.

3. Results and Discussion

To confirm the bonding structure of the as-grown SiN film, XPS measurements were carried out first. Figure 2 shows Si2p XPS spectrum of the as-grown SiN film, which is shown by solid line. Similarly, Si2p XPS spec-
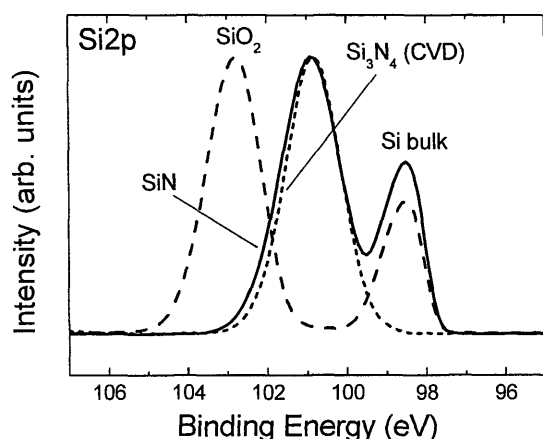
Fig. 2 Normalized Si2p XPS spectrum of SiN film. XPS spectra of thermally grown SiO_2 and CVD Si_3N_4 films are also shown as references.

Fig. 3 N1s XPS chemically shifted (a) peak position and (b) FWHM as a function of N_2 mixing ratio [$N_2/(N_2+Ar)$]. The film growth time was fixed at 120 min, the substrate bias was floating bias, and the working pressure was 0.12 Pa.

tra of thin SiO_2 and thick Si_3N_4 film are also shown in Fig. 2 as references with dash and dotted lines, respectively. The SiO_2 film was thermally grown at 900°C for 10 min to get a thickness of 5.8 nm, and the Si_3N_4 film was commercial film with a thickness of 1000 nm fabricated by thermal CVD at 900°C. For comparison, the three spectra of thin ECR-SiN, SiO_2 and thick Si_3N_4 films are normalized by the intensity of Si2p from the films. The position difference among these three chemically shifted Si2p peaks indicates that the as-grown SiN film fabricated by ECR plasma is nearly stoichiometric and there is no significant Si-O bonding in the film. On the other hand, it was found from Gauss fitting results that the shape of the Si2p peak from the as-grown ECR-SiN film has good symmetry and no hidden peak exists. According to the above analysis, the fabricated film is a pure and nearly stoichiometric SiN film instead of an oxynitride film.

3.1 Effect of N_2 mixing ratio on growth characteristics of SiN film

We have shown from electrical measurements that the N_2 mixing ratio in the plasma has decisive effect on the film quality.[21] When the N_2 mixing ratio was about 60%, the film showed the best electrical properties, including the lowest EOT and low leakage current density at the same irradiation time of 120 min. Here, the SiN film was fabricated at 400°C under a floating bias (+5 V). The effect of N_2 mixing ratio on film quality can be also observed in XPS results. The chemically shifted peak position and the full-width at half-maximum (FWHM) of the N1s signal as a function of N_2 mixing ratio are shown in Fig. 3. In this
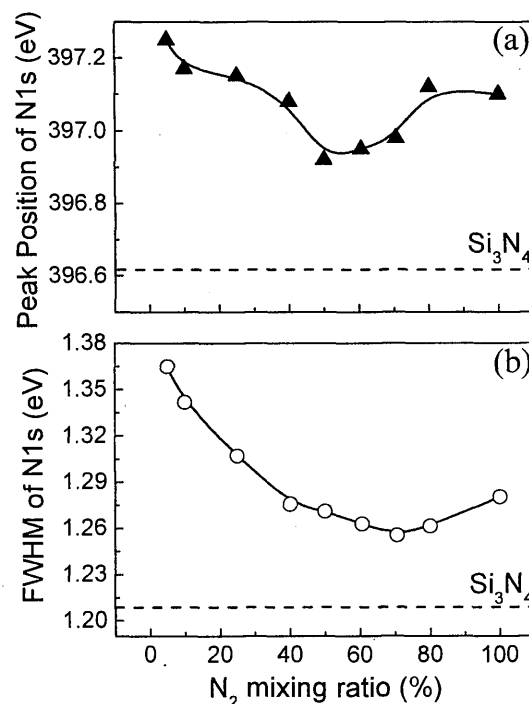
figure, the peak position and FWHM of the N1s signal obtained from the stoichiometric Si_3N_4 film are also shown by dash lines for reference. It can be seen that the SiN film fabricated at the N_2 mixing ratio of approximately 60% is nearest to the stoichiometric structure.

The reason that 60% N_2 mixing ratio is the best condition is found in the XPS spectra of impurities in the film. Two kinds of impurities, N_2 molecules and Ar atoms, are found in the as-grown SiN film. From N1s XPS spectrum, a small peak related to N_2 molecules is found beside the large peak from Si-N bond. From Ar2p spectra, it is confirmed that some Ar atoms are contained in the SiN film. Figures 4(a) and (b) show Ar2p intensities related to Ar atoms and N1s intensities related to N_2 molecules as a function of N_2 mixing ratio, respectively. In Fig. 4(a), with the increase of N_2 mixing ratio, the Ar2p intensities decrease almost linearly. This result should be caused by the decrease of Ar concentration in the mixing plasma. On the other hand, this figure also indicates that the existence of Ar atoms in the SiN film would not affect the film quality greatly. The reason is as follows: there is no Ar atom in the film at 100% point, but Ar atoms obviously exist at 60% point. If the film quality is sensitive to the existence of Ar, the sample at 100% point should have the best film quality. However, the sample at 60% point has the best
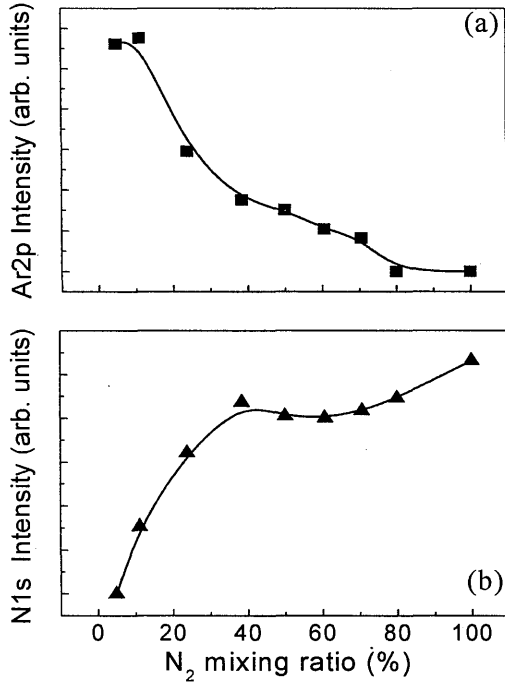
Fig. 4 Ar2p and N1s XPS chemically shifted peaks intensity as a function of N_2 mixing ratio $[N_2/(N_2+Ar)]$. The N1s XPS peak is related to the N_2 molecules in SiN film. SiN film growth conditions: see Fig. 3.

film quality. Thus, it can be concluded that the existence of Ar atoms has no great effect on the film quality.

The dependence of N_2 molecules concentration on the N_2 mixing ratio is shown in Fig. 4(b). The electrical measurement shows that the film quality do not change dramatically when the N_2 mixing ratio is higher than 40%, which means that the N_2^+ concentration in the plasma is almost enough for the Si surface nitridation. The increase of N_2 mixing ratio in the range more than 40% would cause two kinds of effects, one is the increase of the N_2^+ concentration during plasma irradiation, leading to improvement of the film quality; another is incorporation of N_2 molecules in the film. Thus, at 60% point, the most suitable ratio for getting good film quality is achieved. In other words, Ar dilution with suitable mixing ratio would increase nitrogen radical concentration in the Ar/N_2 mixed plasma. In the higher N_2 mixing ratio range more than 60%, too much N_2 molecules cause the degradation of the film quality. Therefore, it can be concluded that N_2 molecules concentration in the SiN film has decisive effect on the film quality.

3.2 Effect of substrate bias on growth characteristics of SiN film

In ECR system, a static electric field is gen-

erated because of the applied magnetic field diverging towards the sample substrate. This electric field accelerates ions and decelerates electrons along the plasma stream from the plasma chamber to the substrate, which induces an effective ion transport to the substrate surface.[22] The substrate surface energy E_S consists of the following two components:[23]

$$E_S = E_{th} + E_i \qquad (1)$$

where E_{th} is the thermal energy component; E_i is the additional energy component supplied to the substrate surface by the impinging plasma ions and is expressed as follows:

$$E_i = K\Gamma_f \varepsilon_i \qquad (2)$$

where K is the kinetic energy conversion coefficient from impinging ions to an Si surface; Γ_f is the ion flux; ε_i is the kinetic energy of an individual ion impinging onto the surface due to the difference between the plasma potential and the substrate bias(V_b). In this study, the substrate temperature and gas pressure are fixed, implying that E_{th} and Γ_f are kept constant. Thus, when a positive substrate bias is applied, E_i decreases and leads to the weak ion irradiation. When a negative bias is applied, E_i increases and leads to the strong ion irradiation. In our ECR equipment, plasma potential was measured as about 20 eV.[24]

Figures 5 (a) and (b) show the film thickness (d), EOT, and refractive index (n) of the SiN film as a function of V_b, where the N_2 mixing ratio was fixed at 60% and the film growth time was fixed at 120 min. The d and n were measured by SE, and EOT was obtained by C-V characteristics. The results can be classified into three types.

Type I result is in the V_b range of less than -10 V. In this range, both of the film growth and plasma etching should occur because of the strong negative bias. However, d of SiN film keeps nearly constant of 3.7 nm even though the negative bias is increased. It is likely that there is a minimum thickness limitation (critical thickness: d_c) caused by homeostasis between the film growth and the plasma etching. This will be discussed in Sec. 3.3. In the same way, n, which is associated with permittivity of SiN film, also keeps constant, and thus a constant EOT can be explained for type I result.

Type III result is in the V_b range of more than +10 V. Similar to type I result, type III result shows a constant d, EOT, and n, although d is a rather high value of about 5.6 nm. The reason is that there is no perceptible plasma etching effect because of the weak ion
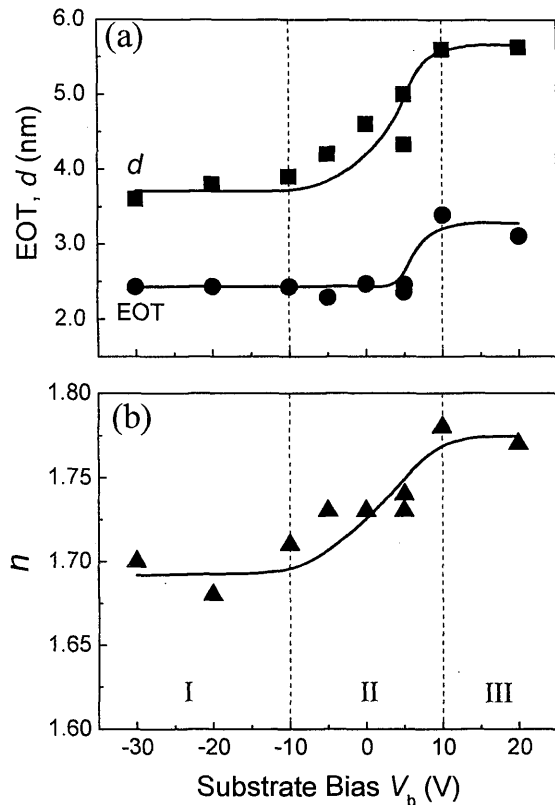
Fig. 5 (a) Physical thickness (*d*) and EOT, (b) refrac-
tive index (*n*) as a function of substrate bias.
The film growth time was fixed at 120 min, the
N₂ mixing ratio was 60%, and the working
pressure was 0.12 Pa.

Fig. 6 The ratio of leakage current density at +1V of
SiN film to that of thermally grown SiO₂ film
with the same EOT as a function of substrate
bias. SiN film growth conditions: see Fig. 5.

irradiation, and the film thickness is controlled
only by the growth rate. Thus, constant *d*, *n*,
and EOT in the type III result can be explained
by the fact that the growth rate is insensitive
to the positive V_b.

Type II result is a transitional one between
type I and type III results. With decreasing V_b
from +10 to -10 V, plasma etching effect be-
comes stronger, while film growth rate keeps
constant or increases a little. Thus, the film
thickness decreases gradually, which seems to
cause the decrease of *n* synchronously. How-
ever, a constant EOT in this range indicates
that the film quality, especially film permit-
tivity, decreases gradually together with de-
creasing V_b from positive to negative. This
speculation can be confirmed by the detailed
study of leakage current density of the film.

Figure 6 shows the ratio of the leakage cur-
rent density at +1V of SiN film to that of
thermally grown SiO₂ film with a given EOT
as a function of V_b. The plasma etching has two
opposite kinds of effects on the film quality, i.e.,
the advantage is to improve the film quality by
breaking some weak Si-N bonds, while the
disadvantage is to degrade the film quality by
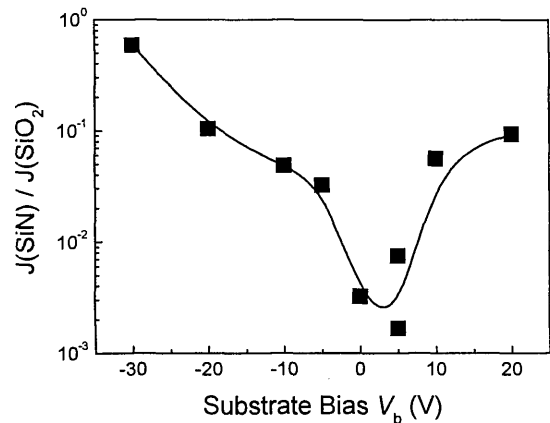introducing some defects into the film. Since

the defects in the film would dramatically in-
crease the leakage current density, there must
be a suitable V_b, which can induce suitable
plasma etching effect to improve the film
quality. It can be seen from Fig. 6 that the ratio
of leakage current density has a minimum
value at a floating bias (+5 V), implying that
the most compact SiN film is obtained at this
point because of the suitable plasma etching
effect. When the V_b is lower than -5 V, too
strong plasma etching causes much defects in
the film, as a result, the leakage current den-
sity increases greatly. When the V_b is higher
than +10 V, although there are no significant
defects caused by plasma etching, some un-
stable Si-N bonds are contained into the film
because of the weak ion irradiation, leading to
the increase of the leakage current density.
Thus, a suitable V_b is carefully selected at
floating bias (+5 V) in this study.

The dependence of the film quality on V_b was
also study by XPS. As shown in Fig. 7, Si2p
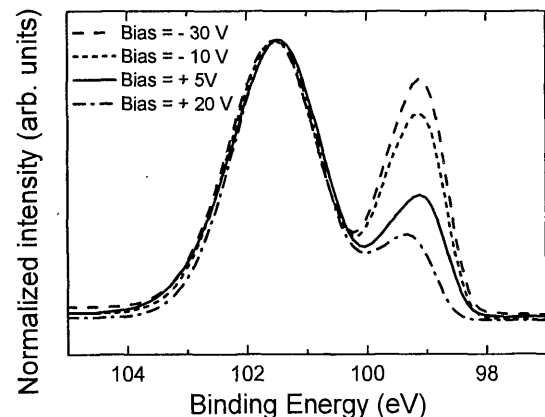XPS spectra of four SiN films grown at differ-

Fig. 7 Normalized Si2p XPS spectra of SiN film fab-
ricated with different substrate bias. SiN film
growth conditions: see Fig. 5.

ent V_b are normalized by the intensity of Si2p from films. The smaller peaks at around 99.2 eV come from Si substrate. It is found that d is very sensitive to V_b, which is similar to the result in Fig. 5. However, the peak position and FWHM of XPS spectra at 101.5 eV are not sensitive to the different V_b. This means that the bonding structure in the film is not greatly affected by the ion irradiation during SiN growth.

3.3 Growth mechanism of SiN film

The dependence of the SiN film thickness on the nitridation time was investigated. The results are shown in Fig. 8, where the film thickness was measured by SE. The nitridation at 400°C was performed under the conditions of N₂ mixing ratio of 60%, V_b of floating bias, and working pressure of 0.12 Pa, while the irradiation time was changed from 30 sec to 120 min. It can be seen from Fig. 8 that the growth characteristics can be divided into two regions of A and B. Region A is the initial stage of SiN film growth, in which the film thickness increases rapidly from zero to about 3.5 nm. Region B is a stable stage, in which the slope in the square root plot of the nitridation time becomes smaller with the increase of the growth time and the final slope is twice smaller than the initial slope in the region B.

The difference between growth rates in regions A and B can be explained by the model in Fig. 9. In region A, the film growth rate is controlled only by chemical surface reaction because the active N₂⁺ particles in the plasma react with the bare Si surface layer directly, as shown in Fig. 9(a). During this short period, the film growth rate is much higher than the plasma etching rate, leading to the rapid in-
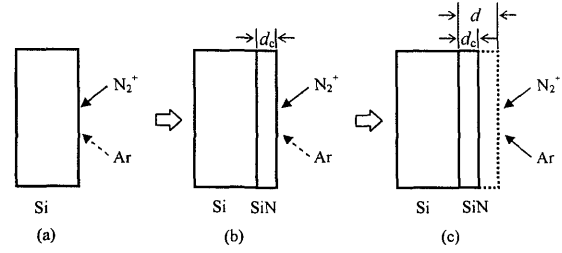
Fig. 9 The model for SiN film growth. (a) stage just after N₂ plasma irradiation, (b) initial and (c) stable stages of the SiN film growth.

crease of d from zero to a critical thickness (d_c), as shown in Fig. 9 (b), where d_c is estimated as 3.5 nm from the result of Fig. 8.

When d becomes thicker than d_c, corresponding to region B in Fig. 8, the growth rate is limited by the diffusion of N₂⁺ into SiN film and the plasma etching effect becomes crucial. With the increase of d, the film growth rate decreases gradually because of the diffusion-limited reaction of N₂⁺, while the plasma etching rate keeps constant. When the film growth rate becomes the same as plasma etching rate, a homeostasis state would be kept and a film growth would be limited, as shown in Fig. 9(c). In fact, the growth rate slope in Fig. 8 becomes smaller and smaller with increasing the growth time. It should be noted that d of SiN film cannot be thinned less than d_c in region B even though the strong ion irradiation is used, because the film growth rate is much higher than the plasma etching rate for d less than d_c.

Furthermore, d in Fig. 5 is also different in the three regions. The d in the type I result (strong ion irradiation) is about 3.7 nm, which is close to a critical thickness d_c. The fact that d kept constant in spite of the increase of negative bias is well understood by the above model. The d in the type II result (moderate ion irradiation) decreases gradually with decreasing V_b from +10 to -10 V, in which homeostasis states are almost satisfied and d depends on the ion irradiation energy, i.e., when the plasma irradiation becomes stronger, d becomes thinner. We believe that this suitable plasma etching is essential for high quality SiN fabrication, because some unstable Si-N bonds in the SiN film fabricated during initial stage can be removed by the plasma etching.
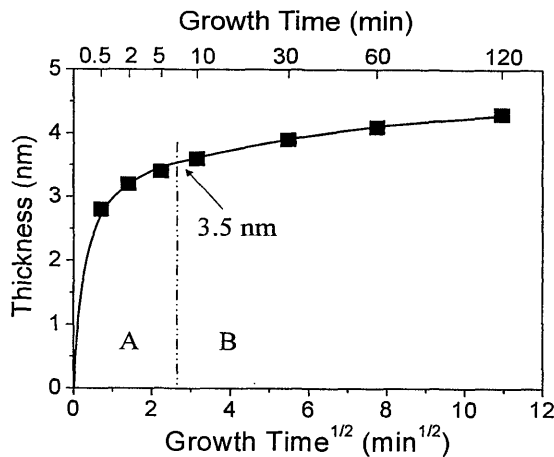

Fig. 8 Growth time dependence of SiN film thickness under the optimized conditions. The N₂ mixing ratio was 60%, substrate bias was floating bias, and the working pressure was 0.12 Pa.

3.4 Structural and electrical properties of SiN film

The HRTEM image of the SiN film is shown in Fig. 10, where the film was formed under
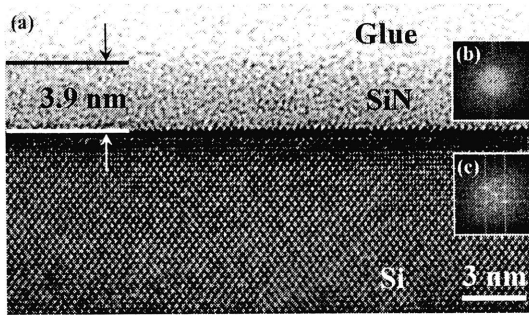
Fig. 10 HRTEM image of SiN film with EOT of 2.46 nm. (a) HRTEM image; (b) and (c) the diffraction patterns of SiN and Si regions, respectively.

optimized conditions for 120 min, which means that the working pressure was 0.12 Pa, the N_2 mixing ratio was 60%, the V_b was floating bias and the substrate temperature was 400°C. The HRTEM image indicates that there are two amorphous regions with different contrasts. The upper region is due to the glue used for sample preparation and the lower region is due to the SiN film. Figures 10(b) and (c) show the diffraction patterns of the SiN and Si regions, respectively; they also show that the SiN film is exactly amorphous and the Si is crystalline. Furthermore, this image shows that the physical thickness of the film is 3.9 nm.

It can be seen from the image that the sample has an atomically flat interface between Si and the SiN film. It can be explained on the basis of the model for SiO_2 film proposed by Toriumi et al [25] that the diffusion of nitrogen radicals in grown SiN is responsible for the flat interface. The model is schematically shown in Fig. 11. According to this model, the nitrogen radicals concentration decreases exponentially in the direction of diffusion from the SiN film to Si and the reaction of nitrogen radicals with Si atoms is very fast. Thus, the Si micro-clusters appeared at the SiN/Si interface because of local Si roughness interact with the nitrogen radicals while other inner Si atoms will not. Therefore, an atomically flat interface
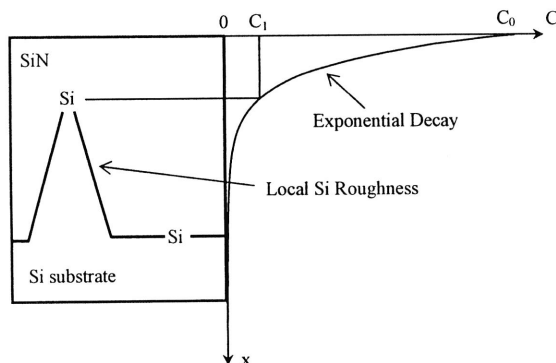
is formed. In other words, the diffusion of the nitrogen radicals plays a "roughness screening" role in forming the flat interface.

Figure 12(a) and (b) show the electrical characteristics of the SiN film formed in N_2/Ar plasma at 60% for 120 min, where EOT was 2.46 nm. The normalized high frequency (10 kHz) $C\text{-}V$ characteristic for the SiN film is shown in Fig. 13(a). As comparison, a calculated ideal $C\text{-}V$ curve is also shown by a solid line. From high − frequency $C\text{-}V$ method, the interface state density at the flat band was estimated as 6.4×10^{11} $eV^{-1}cm^{-2}$, suggesting that very thin SiO_2 interlayer is needed between Si substrate and SiN film to improve the interface quality.

The $J\text{-}V$ curve of the SiN film was also shown in Fig. 12(b). From the figure, the leakage current density was 4.2×10^{-5} A/cm^2 at +1 V, which is more than two orders of magnitude lower than that of thermally grown SiO_2 with a given EOT. It also can be seen that the breakdown happened at 4.8 V, which was corresponding to a breakdown field of 20 MV/cm. Considering the physical thickness of this film, which was obtained from HRTEM image, the permittivity of the SiN film was calculated to be 6.2, which was 1.6 times larger than that of SiO_2.
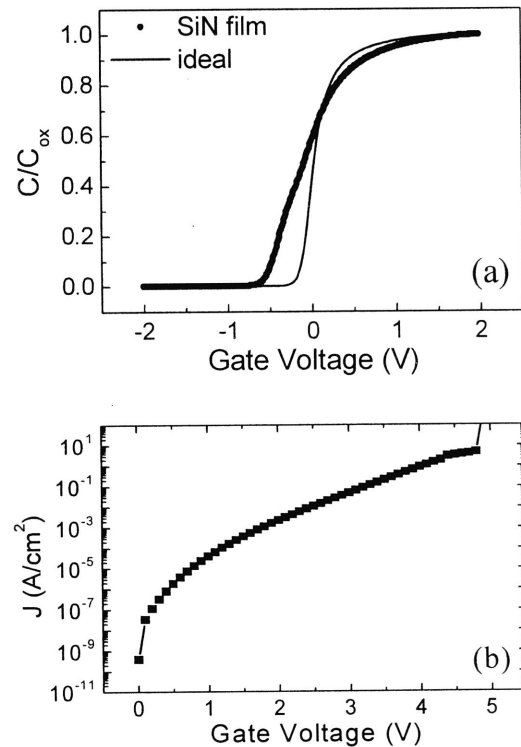

Fig. 11 Model for atomically flat interface formation.

Fig. 12 Electrical characteristics of SiN film with EOT of 2.46 nm. (a) normalized $C\text{-}V$ curve and (b) $J\text{-}V$ curve. The $C\text{-}V$ curve was obtained at a frequency of 10 kHz.

4. Conclusion

An amorphous SiN film has been formed at a low temperature of 400°C by Ar-diluted N_2 ECR plasma. It was found that two experimental parameters, N_2 mixing ratio and substrate bias, have great effect on the structural properties and electrical characteristics of the grown SiN film.

The N_2 mixing ratio strongly affected the SiN film quality. A SiN film having a structure nearest to stoichiometric was obtained with N_2 mixing ratio of 60%, which means Ar dilution with suitable mixing ratio increased nitrogen radical concentration in the Ar/N_2 mixed plasma.

The plasma etching caused by the substrate bias application also affected the SiN film quality. The strong negative and positive biases caused the existence of much defects and weak Si-N bonds in the films, respectively, consequently, the leakage current density increased largely. An optimized substrate bias contributed to depressing leakage current density of SiN film by suitable plasma etching effect. The growth time dependence of SiN film showed rapid growth at the initial stage, followed by the slow growth, and such behaviors could be explained by the homeostasis states between the growth and etching of SiN film.

Two kinds of impurities, N_2 molecules and Ar atoms, were found in the SiN film by XPS measurements. The N_2 molecules concentration in the film had decisive effect on the film quality. The diffusion of Ar atoms into the film was mainly caused by plasma etching effect, but Ar atoms in the films had no great effect on the film quality.

The HRTEM micrograph showed an atomically flat interface of Si and SiN film, and the reason was explained by the reaction of nitrogen radicals with Si micro-clusters.

After 120 min irradiation under the optimized conditions, a SiN film having EOT of 2.46 nm was formed, which showed a leakage current more than 2 orders of magnitude lower than that of thermally grown SiO_2 having the same EOT.

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