Memory effect in silicon nitride deposition using ICPCVD technique

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Abstract
In this study, a plasma-based low-temperature, low-pressure SiN film deposition is investigated for device applications. Ammonia, nitrogen and silane are being used for optimization of the quality of SiN film for device passivation by ICPCVD. Characterization of SiN film is done using elastic recoil detection analysis, AFM, FTIR and ellipsometry. The effect of previous process parameters on subsequent process is called memory effect, which has been investigated by all the characterization techniques. During deposition, this effect has been observed for the same parameters that are used to maintain the stoichiometry of the film. It has been observed that some of the residues of gases used for SiN deposition remain present even after the deposition in the chamber and are carried over for the next deposition process and alter the film property, though parameters such as flow rate, temperature, pressure and time remain fixed. This memory effect alters the film surface roughness and stoichiometry thus affecting device characteristics after passivation.

Keywords Silicon Nitride (SiN) · ICPCVD · PECVD · ERDA · HEMT · Current collapse

Introduction
The silicon nitride dielectric films are being used widely for electrical isolation, mask fabrication and as protection layers in semiconductor device technology [1]. The important and crucial application of these plasma-deposited silicon nitride dielectric films is for passivation of devices [2]. These films can be deposited with different composition and by different techniques as per the requirement. Silicon nitride (Si3N4) is being used in GaAs HEMTs (high-electron mobility transistors) and GaN HEMTs-based MMICs (microwave monolithic integrated circuits) for device passivation. These films reduce the surface trap density of HEMT structure by reducing the surface states at the surface, which is the source of current collapse or knee walkout problems [3, 4]. Silicon nitride (SiN) film composition and quality affect the performance of the device [5]. There are a number of studies reported in the literature for GaN HEMT gate passivation model using PECVD SiN to reduce reverse leakage current [6, 7]. There are very few studies that report results of ICPCVD passivation effect. Chao et al. [8] have reported recently the impact of stress in SiN film deposition by ICPCVD. Memory effect is being very crucial for repeatability and reproducibility of plasma process, but no report is yet published of ICPCVD passivation to the best of our knowledge. The present study reports the detailed systematic study of memory effect of ICPCVD process for SiN passivation [9, 10]. The main aim of the present study is to develop a process free from memory effect to mitigate the problem of current collapse/knee walkout in in-house fabricated GaN HEMTs.

Experiment details
The inductively coupled plasma chemical vapor deposition technique (ICPCVD) based on high-density plasma is used, and we have used Oxford make ICPCVD system for silicon nitride deposition which helps depositing highly dense silicon nitride films with less content of hydrogen percentage compared to conventional PECVD technique [11, 12]. Silane (SiH4) and nitrogen (N2) gases have been used instead of silane and ammonia to minimize the incorporation of hydrogen percentage in the film. SENTECH Adv500 Ellipsometer is used for measuring refractive index and thickness of the
grown films. Films are grown to have stoichiometric silicon nitride with refractive index ~ 2.0 and thickness ~ 1000 Å. To emphasize the memory effect simultaneously, three runs on GaAs wafer for silicon nitride deposition are carried out with fix parameters for sample A, sample B and sample C, i.e., gases flow rate, temperature, pressure, frequency, power and time. Silane at the rate of 140sccm and nitrogen at the rate of 18 sccm are used at pressure ~ 10 mTorr, and temperature ~ 100 °C for 10 min for all the three runs. SiH₄ and N₂ undergo a chemical reaction under the activation action of RF plasma [13]. The possible chemical reaction of SiH₄ and N₂ that takes place in plasma is as follows:

\[
3\text{SiH}_4 + 2\text{N}_2 \rightarrow \text{Si}_3\text{N}_4 + 6\text{H}_2. \tag{1}
\]

The characterization of deposited silicon nitride is carried out using ellipsometric measurement for refractive index and thickness, FTIR for bonding strength and AFM for roughness of the deposited film, ERDA (elastic recoil detection analysis) for investigating quantitatively the proportion of silicon, nitrogen and hydrogen present in the deposited films [14, 15].

Elastic recoil detection analysis (ERDA) is a technique specially suited for depth profiling of light elements, which overcomes the limitations of Rutherford backscattering (RBS) technique [16].

High-energy heavy ions can induce structural and compositional changes in the material through which they pass. The study of high-energy ions induced compositional changes in materials can be carried out using ERDA. In our case, a telescope detector was used to identify different recoils originating from a sample. Mostly, it comprises two detectors named as $\Delta E$ and $E_{\text{rest}}$ sub-detectors and therefore called as $\Delta E – E$ detector telescope. The recoils entering the detector lose a fraction of their energy in $\Delta E$ detector and the rest of it in $E_{\text{rest}}$ detector. Energy lost in the first detector was found to be proportional to $MZ^2$, where $M$ and $Z$ are the mass and atomic number of the recoil particle, respectively, which allows for the identification of elements.

### Result and discussion

#### Ellipsometry

Refractive index measurements show variation in index for the first sample to third sample, which was grown one after another under the same conditions. The refractiveindex was found to vary from ~ 1.89 to ~ 2.0, while thickness was almost ~ 100 nm. The effect on the refractive index is attributed to the foreign species like carbon and oxygen present in the chamber when earlier depositions have been carried out.

The refractive index and thickness are measured by laser ellipsometer using laser radiation of 632 nm wavelength. In our case, the refractive index and the thickness of Si₃N₄ thin films deposited on GaAs wafers are measured. For sample A, the measured values for refractive index and thickness are ~ 1.89 and ~ 1050 Å, respectively. For the next two samples, all the deposition parameters are kept the same and clean GaAs wafers are used for silicon nitride deposition. The variation in refractive index and thickness is determined. Sample B is found to have refractive index ~ 1.95, whereas sample C had refractive index ~ 2.0. Slight change in thickness is also observed with 1150 Å for sample B and 1100 Å for sample C.

#### FTIR spectra

The FTIR spectra have been used to determine the chemical bonding among the various elements present in silicon nitride thin films. Figure 1 shows the FTIR spectra for silicon nitride films in the range of 400–6500 cm⁻¹; these films are deposited by ICPCVD at 100 °C. The absorption peaks are observed at 830 cm⁻¹, in all the films, referring to the stretching vibration mode of the Si–N bond. In addition to above peaks, we observed the Si–H wagging mode near 2180 cm⁻¹ and N–H stretching mode around 3340 cm⁻¹. Slightly higher amplitude at these wave numbers for corresponding peaks is also observed, and the positions of the corresponding absorption peaks of the films may be due to change in stoichiometry of the films [17, 18].

#### AFM measurement

In order to understand the topography of deposited thin films, atomic force microscopy (AFM) studies are carried out. The AFM images shown in Fig. 2 reveal variation in grain size on the surface of silicon nitride films. The scanned regions in each case are taken as $2 \times 2 \mu$m² and $5 \times 5 \mu$m². The surface roughness of silicon nitride thin films from these AFM measurements is found to be 0.9 nm, 0.85 nm and 0.80 nm, respectively, for the samples A, B and C. It can be
Fig. 2 AFM images of samples A, B and C showing different grain sizes
said that the films deposited by ICPCVD have larger grain size and surface roughness initially. However, the reduced surface roughness is observed when the process is stabilized which may have led to better stoichiometry of the film.

**ERDA technique**

ERDA analysis shows the elemental concentration depth profiles of the deposited Si$_3$N$_4$ films [19]. Ag$^{7+}$ is used to irradiate the sample at 100 MeV energy for 30 min. The area of the irradiated spot was calculated and is found to be 0.0175 cm$^2$. CANDLE software is used to count the total number of blocks in the samples. These blocks are then further divided into approximate blocks of 100, and the total charge, charge per block, Si, N and H values are determined. These are shown in Fig. 3.

The total number of ions was calculated using the following formula:

$$\text{No. of incidentions} = \frac{[\text{current integrator count (C)}]}{7 \times 1.6 \times 10^{-19}} \times \frac{FCLS1}{\text{current}}.$$  \hspace{1cm} (2)

Fluence is given by:

$$\text{Fluence} = \frac{\text{total no. of ions}}{\text{area of the spot}}.$$  \hspace{1cm} (3)

The fluence versus concentration graphs in Fig. 4 shows the areal density of Si, N and H in the samples A, B and C. This clearly shows the lowering of hydrogen percentage in sample C compared to sample B and in sample B lower to sample A.

The optimized silicon nitride film was deposited as passivation layer on GaN HEMT devices to mitigate of current...
collapse and knee walkout problem as GaN HEMT devices are severely affected with these problems. The current voltage characteristics in pulse mode shown in Fig. 5 compare the results of device before and after passivation using the optimized silicon nitride film. The current recovery measured here was more than 95% with negligible knee walkout compared to DC I–V characteristics.

Conclusions

We have presented a systematic experimental analysis of silicon nitride films deposited by ICPCVD technique. The study shows variation in the composition and quality of the films even with the same deposition parameters. This may be attributed to the residue remained in the deposition chamber and which affect the very quality of the film until stabilized. Elliposemetric measurements have suggested about the film refractive index and thickness, which can be clearly related to density of the film. Lower refractive index films are found to have higher etch rate compared to high refractive index films. ERDA measurement and calculations confirmed the percentage of hydrogen content in the films, and the variation in the refractive index was verified by ellipsometric measurements results. This hydrogen content is initially high, whose incorporation may be attributed to the presence of residuals in the chamber during deposition. Hence, conditioning and stabilization of silicon nitride films are necessary before it can be used for any device application.

Fig. 4 Analysis for composition in samples A, B and C for Si, N, and H versus fluence
Fig. 5  I–V characteristics (Pulse I<sub>d</sub>–V<sub>d</sub>) of GaN HEMT comparing pulse mode current–voltage before and after optimized silicon nitride passivation

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