

Paper

Quadrupole SIMS Analysis of Si Concentration in GaN Layers by a Molecular Ion Detection with a Minor Isotope

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The optimal condition suitable for using a quadrupole SIMS (Q-SIMS) instrument to measure the Si concentration in GaN layers for both selectively isotopic Si-implanted GaN and epitaxially Si-doped GaN was presented. The detection of $^{30}\text{Si}^{14}\text{N}^-$ molecular ions under Cs^+ primary ion bombardment realized the best dynamic range and the lowest detection limit for Q-SIMS analysis of Si in GaN layers. The detection limits of selectively isotopic ^{30}Si and the total amount of Si naturally occurring isotopes in GaN layers can reach $2 \times 10^{14} \text{ cm}^{-3}$ and $8 \times 10^{15} \text{ cm}^{-3}$, respectively. We concluded that a Q-SIMS instrument was applicable for the analysis of the Si depth distribution in GaN layers.

1. Introduction

Recently, gallium nitride (GaN) -based metal-oxide-semiconductor field-effect transistors (MOSFETs) have begun to be studied and developed for the application to fail-safe high-power switching devices [1-6]. Donor doping with Si is the most common method for achieving *n*-type conducting GaN. Since the control of carrier concentration in *n*-type GaN layers is very important for off-state breakdown voltage and on-state resistance characteristics, the quantitative analysis of Si concentration in *n*-type ohmic contact and reduced surface field (RESURF) GaN layers by secondary ion mass spectrometry (SIMS) measurements is required [7]. Generally, a magnetic sector SIMS instrument is capable of a lower detection limit and higher mass resolving power, whereas a quadrupole SIMS (Q-SIMS) instrument provides better depth resolution for thin film measurement. When a Q-SIMS instrument is used for Si depth profiling in thin GaN layers, Q-SIMS users have been faced with a serious problem that it is impossible to discriminate the mass interferences of $^{12}\text{C}^{16}\text{O}$ and $^{14}\text{N}_2$ molecular ions from ^{28}Si atomic ions [8], leading to the high background of ^{28}Si in GaN layers.

Our final goal for this study is to solve the problem about mass interferences and to perform a high-depth resolution analysis for depth

profiling of the Si in the GaN layer by a Q-SIMS instrument. In this paper, the measurement conditions suitable for quadrupole SIMS analysis of the Si concentration in GaN layers are systematically studied.

2. Experimental

The 3.5- μm -thick undoped GaN epitaxial layers grown on sapphire substrates by metalorganic vapor phase epitaxy (MOVPE) were used. Three kinds of Si isotopic ion implantation samples were prepared, as shown in Fig. 1(a). The ^{28}Si , ^{29}Si and ^{30}Si isotope ions were implanted into

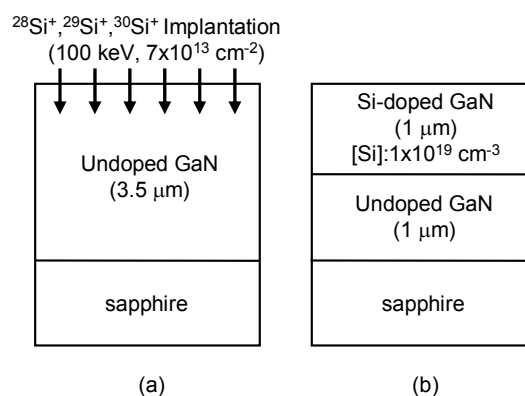


Fig. 1 GaN samples used in this study.

undoped GaN layers with a nominal dose of $7 \times 10^{13} \text{ cm}^{-2}$ with 100 keV of implantation energy and at a 7° implantation angle.

From the viewpoint of a practical SIMS analysis of Si depth profiling in GaN, it is necessary to measure the total amount of Si naturally occurring isotopes (^{28}Si : 92.2%, ^{29}Si : 4.7%, ^{30}Si : 3.1%) in Si-doped GaN epitaxial layers. In addition, analyzing the background level of Si impurities in undoped GaN epitaxial layers is also important. For this investigation, a 1- μm -thick Si-doped GaN/1- μm -thick undoped GaN layer structure sample was also prepared, as shown in Fig. 1(b). The Si donor concentration was about $1 \times 10^{19} \text{ cm}^{-3}$.

SIMS measurements were carried out with the quadrupole based instrument (PHI-6650, Physical Electronics, Inc.). Negatively charged secondary ions such as Si^- , SiN^- and GaN^- were collected using Cs^+ primary ions at the incident angle of 60° . The GaN^- signal was taken as the matrix reference ion. All GaN layers were sputtered with 5 keV Cs^+ primary ions scanning over an area of $200 \mu\text{m} \times 400 \mu\text{m}$. The electron beam irradiation was applied for charge neutralization.

Sputter rates were determined by measuring the crater depth with the Talystep profilometer (Taylor Hobson).

In order to investigate the mass interference in detail, the cemea IMF-6f magnetic sector SIMS measurements were also taken in high mass

resolution mode with $M/\Delta M \sim 4000$ of mass resolving power.

3. Results and Discussion

3.1 Secondary ion species.

Figure 2 compares two depth profiles for $^{28}\text{Si}^-$ atomic and $^{28}\text{Si}^{14}\text{N}^-$ molecular ions. The secondary ion intensity of $^{28}\text{Si}^-$ atomic ions is not so high for a Q-SIMS instrument. The secondary ion intensity of $^{28}\text{Si}^{14}\text{N}^-$ molecular ions was approximately 40 times higher than that of $^{28}\text{Si}^-$ atomic ions. Additionally, the dynamic range of Si became large. Therefore, the SiN^- molecular ions have been selected to improve the secondary ion intensity and the dynamic range of Si in GaN. Takakuwa-Hongo and Tomita reported a similar result that the detection sensitivity of carbon in GaN using the CN^- molecular ions had improved [9].

3.2 Selectively Isotopic Si-implanted GaN.

Figure 3(a) shows the depth profiles of ^{28}Si concentration in GaN monitored by $^{28}\text{Si}^{14}\text{N}^-$ ions at the offset bias of 0 V and 20 V for the ^{28}Si -implanted GaN. At the offset bias of 0 V, a background level of Si in GaN is very high.

To evidence the origin of this high background, the Si-doped GaN layer was measured under high mass resolution mode with the magnetic sector SIMS instrument. The high resolution mass spectrum around m/z 42 had three clear peaks: one is for $^{28}\text{Si}^{14}\text{N}^-$, second is for $^{12}\text{C}^{14}\text{N}^{16}\text{O}^-$ due to an unintentional impurity such as C and O, and the other is for $^{14}\text{N}_3^-$ due to a matrix element. Therefore, it is confirmed that the high background level for monitoring the $^{28}\text{Si}^{14}\text{N}^-$ molecular ions by Q-SIMS measurements was due to the strong mass interferences.

In case that the energy distribution of secondary ions between the ion of interest and ions of interference is different, an energy offset method is available. The background level of ^{28}Si in GaN can reach $1 \times 10^{17} \text{ cm}^{-3}$ when the offset bias of 20 V is applied, as shown in Fig. 3(a). The energy offset technique is useful in reducing the mass interference from other ions. However, this background level is not quite enough.

Figure 3(b) indicates the depth profiles of ^{29}Si concentration in GaN monitored by $^{29}\text{Si}^{14}\text{N}^-$ ions at the offset bias of 0 V and 10 V for the ^{29}Si -implanted GaN. We also found that the energy offset technique is effective in eliminating the mass interference from the other ions, as

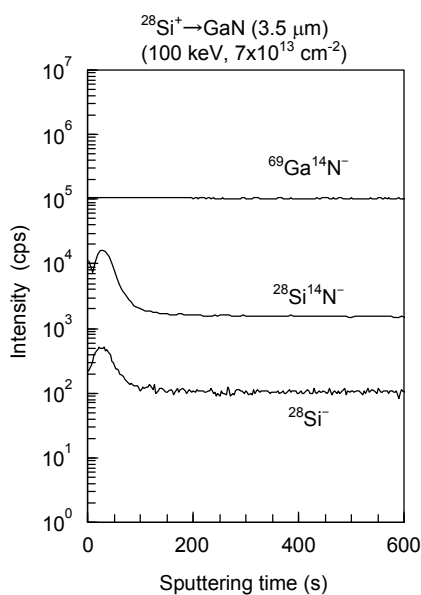


Fig. 2 The secondary ion intensity of $^{28}\text{Si}^-$ atomic and $^{28}\text{Si}^{14}\text{N}^-$ molecular ions in the ^{28}Si -implanted GaN layer.

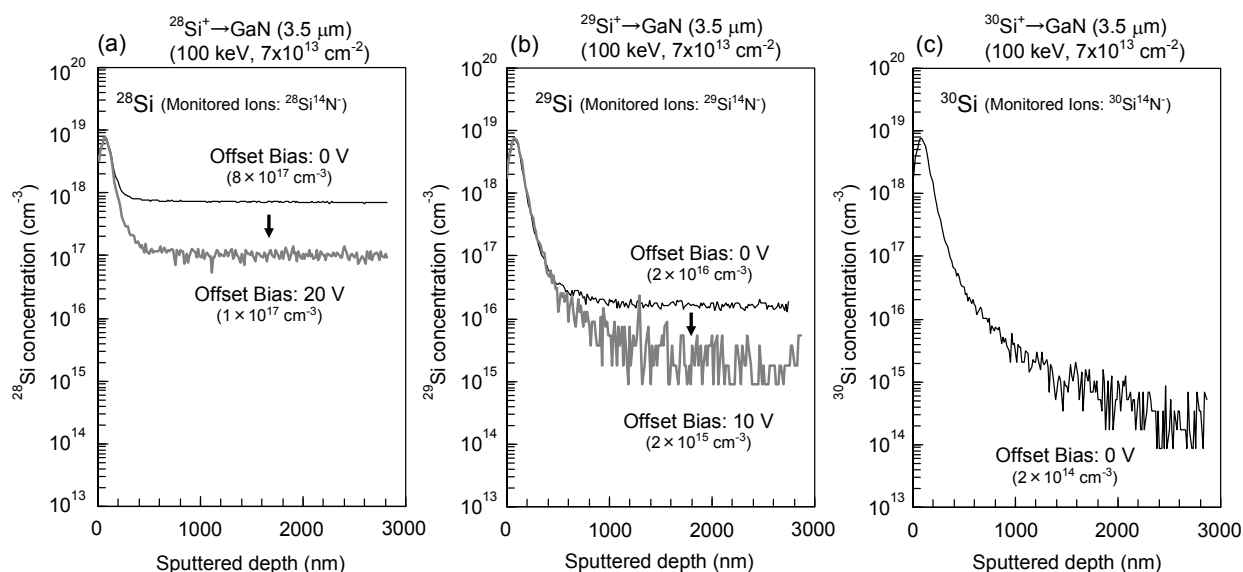


Fig. 3 SIMS depth profiles of (a) ^{28}Si -, (b) ^{29}Si - and (c) ^{30}Si -implanted (100 keV , $7 \times 10^{13}\text{ cm}^{-2}$) GaN layers at different offset bias.

shown in Fig. 3(b). The detection limit of ^{29}Si in GaN can reach $2 \times 10^{15}\text{ cm}^{-3}$ at the offset bias of 10 V. No further improvement of the detection limit was observed when the offset bias was applied more than 10 V owing to a loss of the signal intensity of $^{29}\text{Si}^{14}\text{N}^-$.

The depth profile of the ^{30}Si concentration in GaN monitored by $^{30}\text{Si}^{14}\text{N}^-$ ions at the offset bias of 0 V for the ^{30}Si -implanted GaN is shown in Fig. 3(c). The $^{30}\text{Si}^{14}\text{N}^-$ molecular ions give a more dynamic range and no background signal levels. The detection limit of ^{30}Si as low as $2 \times 10^{14}\text{ cm}^{-3}$ can be reached. For Q-SIMS analysis of ^{30}Si in GaN, the energy offset technique had no effect on improving the detection limit of ^{30}Si monitoring $^{30}\text{Si}^{14}\text{N}^-$ because a loss of the signal intensity of $^{30}\text{Si}^{14}\text{N}^-$ leads to degrading the detection limit.

These results revealed that, if the selectively isotopic ^{30}Si -implanted GaN samples are prepared, the $^{30}\text{Si}^{14}\text{N}^-$ molecular ion detection at the offset bias of 0 V under Cs^+ primary ion bombardment is very effective in characterizing the channeling profile of the Si ion implantation process and the Si redistribution after the annealing process [7].

3.3 Naturally Occurring Isotope Si-doped GaN.

From a practical prospective, it is essential to measure the total amount of Si naturally occurring isotopes ($^{28}\text{Si}+^{29}\text{Si}+^{30}\text{Si}$) in Si-doped GaN epitaxial layers and to analyze the background level of Si impurities in undoped GaN epitaxial layers.

When the naturally isotopic Si concentration in GaN epitaxial layers is quantified, we must correct the measured intensity for SiN^- molecular ions with the isotope abundances of both Si (^{28}Si , ^{29}Si and ^{30}Si) and N (^{14}N and ^{15}N).

When the Si concentration by monitoring $^{29}\text{Si}^{14}\text{N}^-$ molecular ions is quantified, it is necessary to correct the N isotopic abundances (^{14}N : 99.63%, ^{15}N : 0.37%). Because of the same nominal mass m/z 43, there is mass interference between $^{29}\text{Si}^{14}\text{N}^-$ and $^{28}\text{Si}^{15}\text{N}^-$ molecular ions. The ratio of isotope abundances of $^{28}\text{Si}^{15}\text{N}$ to $^{29}\text{Si}^{14}\text{N}$ ($^{28}\text{Si}^{15}\text{N}/^{29}\text{Si}^{14}\text{N}$) is $(0.922 \times 0.0037) / (0.047 \times 0.9963) \sim 1/14$. Therefore, to avoid a slight overestimation of Si concentration obtained by monitoring $^{29}\text{Si}^{14}\text{N}^-$ ions, the contribution of the $^{28}\text{Si}^{15}\text{N}^-$ signal intensity has to be subtracted from the measured signal intensity of m/z 43, $^{43}(\text{SiN})^-$.

Similarly, for the detection of the $^{30}\text{Si}^{14}\text{N}^-$ molecular ions, it should be noted that the signal intensity of $^{30}\text{Si}^{14}\text{N}^-$ interfered with that of $^{29}\text{Si}^{15}\text{N}^-$. However, the interference of $^{30}\text{Si}^{14}\text{N}^-$ with $^{29}\text{Si}^{15}\text{N}^-$ is normally low because the $^{29}\text{Si}^{15}\text{N}/^{30}\text{Si}^{14}\text{N}$ isotope abundances ratio of $(0.047 \times 0.0037) / (0.031 \times 0.9963) \sim 1/177$ is small. Therefore, the influence of the $^{29}\text{Si}^{15}\text{N}^-$ interference ion on the $^{30}\text{Si}^{14}\text{N}^-$ can be negligible.

Figure 4 depicts depth profiles of the total Si concentration monitored by $^{28}\text{Si}^{14}\text{N}^-$, $^{29}\text{Si}^{14}\text{N}^-$ and $^{30}\text{Si}^{14}\text{N}^-$ molecular ions that provide the highest dynamic range for each offset condition used. A

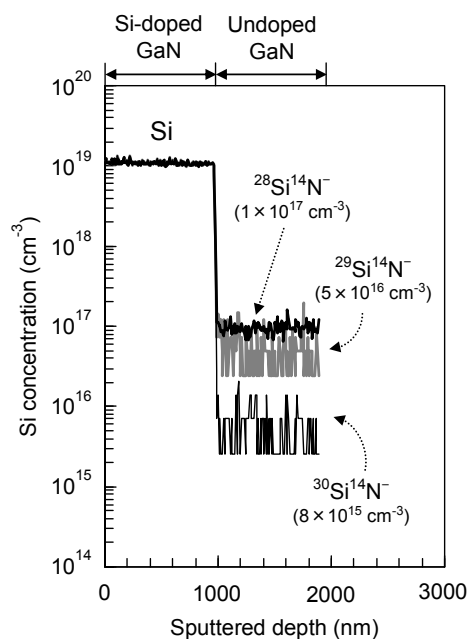


Fig. 4 Si depth profiles in the Si-doped/undoped GaN epitaxial layer acquired by selected molecular ions with the largest dynamic range for each measurement condition.

comparison of the Si concentration in GaN using different isotopic molecular ions was made on Si-doped GaN/undoped GaN epitaxial layers. The depth profile for monitoring $^{30}\text{Si}^{14}\text{N}^-$ molecular ions realizes the best dynamic range and the lowest detection limit ($8 \times 10^{15} \text{ cm}^{-3}$) for the total amount of Si naturally occurring isotopes in GaN layers.

Through this paper, we indicate that the detection of $^{30}\text{Si}^{14}\text{N}^-$ molecular ions provides a hope for the analysis of the Si depth distribution in both selectively isotopic ^{30}Si -implanted GaN layers and naturally occurring isotope Si-doped GaN layers by a Q-SIMS instrument. However, our study is not complete yet. Furthermore, additional experiments are required to develop the strong points of a Q-SIMS such as high depth resolution and thin film measurements.

4. Conclusion

The optimal condition suitable for the Q-SIMS analysis of the Si concentration in both selectively isotopic Si-implanted GaN layers and naturally occurring isotope Si-doped GaN layers was presented. The detection of $^{30}\text{Si}^{14}\text{N}^-$ molecular ions under Cs^+ primary ion bombardment realized the best dynamic range and the lowest detection limit for Si concentration in GaN layers by a Q-SIMS measurement. The detection limits of selectively isotopic ^{30}Si and the total amount of Si naturally occurring isotopes in GaN layers can reach $2 \times 10^{14} \text{ cm}^{-3}$ and $8 \times 10^{15} \text{ cm}^{-3}$, respectively. We concluded that a Q-SIMS instrument was applicable for the analysis of the Si depth distribution in GaN layers.

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