

WHITE PAPER

Measurement of Low Level Nitrogen in Silicon Carbide Using SIMS

ABSTRACT

Today's state of the art silicon carbide (SiC) growth can produce semi-insulating crystals with a background doping around 5×10^{15} atoms/cm³ or lower. It is essential to have an accurate tool with low enough detection limit to measure low level nitrogen concentration. Current SIMS detection limit of low E15 atoms/cm³ will provide accurate determination for nitrogen doping level of 5×10^{16} at/cm³ or higher. In order to determine the lower nitrogen concentration, it is necessary to provide better detection limit and to remove the contribution of background nitrogen properly. The "raster changing" method provides an accurate way to determine and remove contribution of background nitrogen to the signal because secondary ion intensities and matrix ion intensities can be analyzed at same location of sample by changing primary beam raster size during a profile. We performed experiment using these techniques and were able to determine nitrogen concentration of 3.5×10^{15} atoms/cm³ in silicon carbide with good precision.

INTRODUCTION

Due to its unique capabilities of high detection sensitivity for a variety of elements under depth profiling mode, Secondary Ion Mass Spectrometry (SIMS) is an essential tool for characterization of dopants and impurities in SiC material.

Nitrogen, which is a shallow donor in SiC, is always present as trace impurity in all SiC wafers and epi-layers. Depending on the design and its operation, a SiC growth system can contain significant amounts of nitrogen, which will lead to a fluctuating nitrogen residual doping in the grown material. A low and controlled nitrogen background is necessary for a reproducible growth of semi-insulating wafers and low doped epi-layers. SIMS feedback of nitrogen in these crystals can provide understanding of the compensation mechanism and process conditions.

For many years, SIMS has been used routinely to determine nitrogen concentration at level of E17 to E19 atoms/cm³ with good precision. Today's state of the art SiC growth can produce semi-insulating crystals with a background doping around 5×10^{15} atoms/cm³ or lower. It requires SIMS to have better precision to measure low level nitrogen concentration.

With upgraded SIMS instrument (improved vacuum and better primary beam intensity) and improved analysis protocol, we can now achieve N detection limit of $2-5 \times 10^{15}$ atoms/cm³ routinely while maintain excellent depth resolution (Fig 1). While these detection limits are very good, they are not sufficient to provide a routine way for an accurate measurement of low level ($< 5 \times 10^{15}$ atoms/cm³) nitrogen

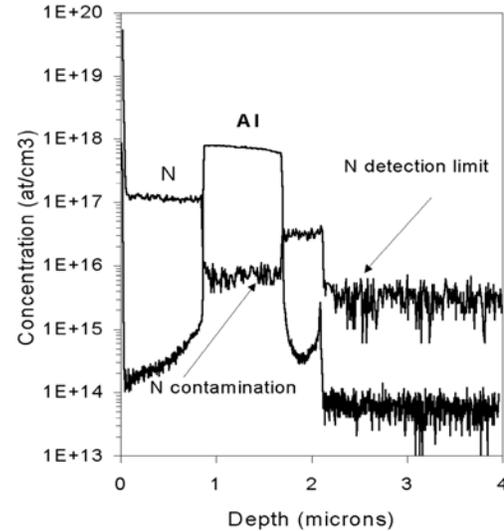


Figure 1: The N detection limit is 3×10^{15} atoms/cm³. Slight amount of N contamination in Al doped layer.

concentration in silicon carbide. A simple background removal at these background level ($2-5 \times 10^{15}$ atoms/cm³) can introduce large and un-defined error. An extremely low nitrogen background ($6-8 \times 10^{14}$ atoms/cm³) is achievable, but is very time consuming and can not be guaranteed at all the times.

To determine low nitrogen concentration with adequate precision, it requires [1] (a) better Signal-to-Noise ratio; (b) a proper way to subtract background contribution from the measured signal because background contribution can change over time, and can also vary from location to location in SIMS sample holder. To solve these issues, we applied SIMS "raster changing" technique on silicon carbide sample with low level of nitrogen concentration ($< 5 \times 10^{15}$ atoms/cm³) to determine the nitrogen concentration in the sample and to verify the precision of the results. The "raster changing" method provides an accurate way to determine and remove contribution of background nitrogen to the signal, because secondary ion intensities and matrix ion intensities can be analyzed at same location of sample by changing primary beam raster size during a profile [2]. The disadvantage of this technique is that it can be used only on uniformly doped sample or in the region of depth that concentration is uniform.

EXPERIMENTS

The experiments were performed on CAMECA IMS-4f double focusing magnetic sector instruments equipped with oxygen and/or Cs primary beam sources. The sample was sputtered by a focused

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Cs+ primary ion beam, which is rastered over a square area. Beam current used in the analysis are 200 to 300 nA. Secondary ions formed during the sputtering process are accelerated away from the sample surface by a sample voltage of - 4500 V. Secondary ions were energy separated by an Electrostatic Analyzer and mass separated based on their mass/charge ratio by a Magnetic Mass Analyzer. After passing through the analyzers, the ions are detected using either a Faraday cup (Matrix ions) or an Electron Multiplier (nitrogen ions). The SiC sample used in this study has low level of nitrogen uniform through out the sample.

RESULTS & DISCUSSION

First, the nitrogen concentration in the test sample was determined using traditional background subtraction method. With a brand new Cs source and a few days pumping with sample in UHV chamber, we were able to achieve nitrogen background of 5e14 atoms/cm3, determined from measurement of a piece of un-doped SiC epi sample that was loaded together with the test sample. We determined the nitrogen concentration in this test sample as 3.3E15 atoms/cm3. The precision of this value is ±14% (1) based on repeat measurements and sample and background. With raster changing technique, the nitrogen concentration [N] in the SiC sample and corresponding contribution of nitrogen from instrument background [NB] can be calculated using [2],

$$[N] = \text{RSF} \times (\text{IN1}-\text{IN2})/(\text{IM1}-\text{IM2})$$

$$[\text{NB}] = \text{RSF} \times (\text{IN}/\text{IM}) - [\text{N}]$$

assuming the adsorption rate of nitrogen on sample surface is constant. Where RSF is Relative Sensitivity Factor determined from nitrogen implant standard in SiC ; IN1 and IN2 are nitrogen secondary ion intensities under two different raster sizes, and IM1 and IM2 are corresponding matrix secondary ion intensities under

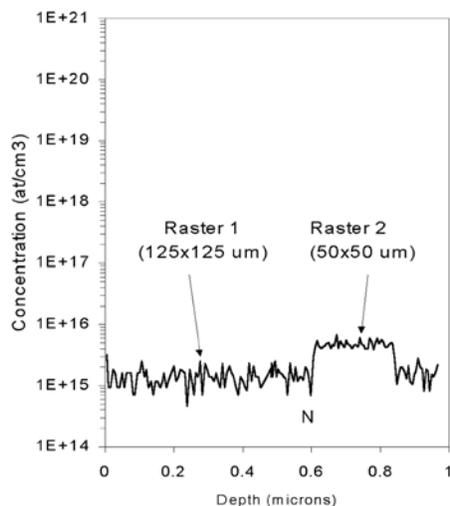


Figure 2: Raster change during N profile. The N shown is quantified at 50x50 µm raster.

Results of nitrogen concentration in the sample and background nitrogen levels are listed in the table below:

Measurement	1	2	3
[N]	3.2E15	3.4E15	3.8E15
[NB]	4.9E15	1.1E15	3.3E15

these two different raster sizes.

In this experiment, nitrogen concentration was determined from nitrogen (N+13C)- and matrix (Si)- secondary ion intensity at 125 µm x 125 µm raster and 50 µm x 50 µm raster (Fig. 2). Then we applied raster changing technique on this sample at different background level ranging from 1e15 atoms/cm3 to 5e15 atoms/cm3.

The average nitrogen concentration determined from three measurements is 3.5E15 atoms/cm3 with precision of ±9% (1). The results show very good agreement with the data previously obtained with tremendous amount of more effort. Results also demonstrated that low level nitrogen can be measured with good precision, even at relatively high nitrogen background level, using raster changing technique.

CONCLUSION

We have demonstrated that SIMS raster changing technique can be used to determine low level nitrogen in silicon carbide sample with good precision. The measurement eliminated the problems associated with the traditional background subtraction method can be used at relatively higher nitrogen background conditions. But this technique is limited to bulk sample or thick layers (>2 µm).

ACKNOWLEDGEMENT

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- [2] A. Ishitani, et al., in Proceedings of the international Conference on Materials and Process Characterization for VLSI (ICMPC'88), 1988, p. 124.