Analysis of $Al_xGa_yIn_zN$ Semiconductors

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Abstract Secondary ion mass spectrometry is an excellent technique to characterize the dopants, impurities, and composition of AlGaInN materials and devices used in optoelectronics. The technique can be utilized to control of materials purity and doping, determine growth rate and composition, as well as impart the structure of finished laser or light emitting diode devices. secondary ion mass spectrometry is thus a powerful tool for failure analysis, reverse engineering, and concurrent engineering.

1 Introduction

Group Ⅲ-nitrides have recently been the subject of intense research on account of their promising applications in blue and ultraviolet optoelectronic devices as well as microwave and electronic devices[1,2]. The growth of epitaxial layers by metal-organic chemical vapor deposition (MOCVD) and molecular beam epitaxy (MBE) requires tight control on purity, doping, alloy composition, thickness, and interface quality. Secondary ion mass spectrometry (SIMS) is an excellent characterization technique due to its ability to depth profile with high sensitivity and good depth resolution[3]. SIMS complements electrical techniques as it detects dopants regardless of their site in the crystal lattice and provides a wealth of information to materials scientists and device engineers, e.g. in the study of impurity activation and dopant diffusion.

2 Experimental

SIMS measurements were performed with a magnetic sector based instrument (Cameca IMS-4f) and quadrupole based instrument (PHI-6600). These two instruments are equipped with dual cesium and oxygen primary ion sources and complement each other in terms of performance. Depth profiles were acquired on the Cameca IMS-4f using 8 keV primary O_2^+ ions with positive SIMS (monitoring positive secondary ions), 5.5 keV Cs^+ ions with positive SIMS (monitoring CsM^+ ions where M is the element of interest), or 14.5 keV Cs^+ ions with negative SIMS (measuring negative secondary ions). The primary bombardment energy varied from 1 keV to several keV for the PHI-6600. The primary beam was rastered over a square region 50 to 125 micrometers on one side, and secondary ions were collected from the central region of the sputtered craters by using a physical aperture 30 μm in diameter (Cameca IMS-4f) or electronic gating (PHI-6600). The sputtering rates were determined by measuring the analytical crater depths with a Tencor P10 stylus profilometer. The Ⅲ-nitride samples were grown by MOCVD. The reference materials were implanted with known doses of the elements of interest including Si, Mg, Zn, Cd, Se, B, H, C, O, Cl, Fe, Mo; Ni, Cu and Mn.

3 Results & Discussion

The ion yield trends in AlGaInN materials have been studied in details[4]. A better understanding of the ion yield systematics and improvement in the experimental techniques have enabled quantitative characterization of Ⅲ-nitride materials and devices by SIMS. Fig.1 illustrates the role of SIMS in a typical GaN MOCVD light emitting diode (LED) process. The applications include the depth profiling of dopants (Mg, Zn, and Si), common impurities (atmospheric and metallic ions), and compositions. These specific applications will be described in the following sections and a measurement on a finished LED device will also be included to demonstrate the latest attempt in using SIMS in the fields of failure analysis, reverse engineering, and concurrent engineering.

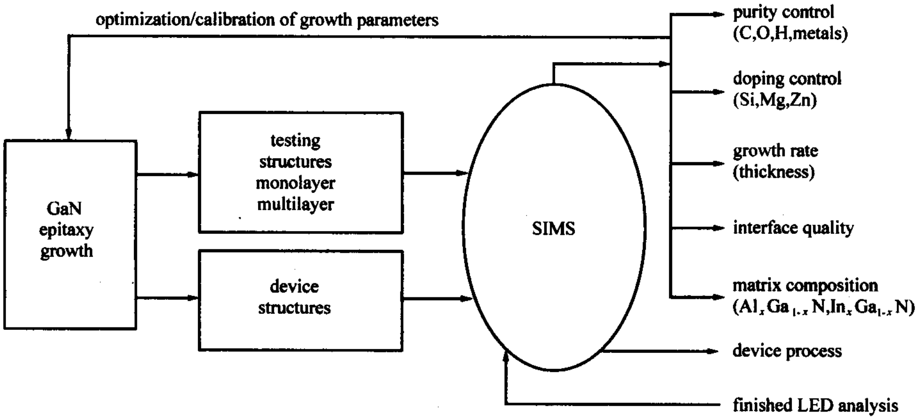

Fig.1 SIMS applications to the characterization of GaN based materials

3.1 Dopant control

SIMS offers good detection sensitivity, and the typical detection limits of the important elements are exhibited in Table 1. The elements Si, Zn and Mg are currently used as n-and p-dopants for the GaN system. For doping control, there is a need for an accurate and rapid technique to calibrate the dopant sources, and when using ion-implanted standards, SIMS can provide a means of measuring the concentration of impurity with an accuracy of better than 10%. By comparing the atomic concentration of the dopant with electrical carriers determined by Hall measurements and *C-V* profiling, the doping efficiency can be readily deduced. Moreover, with a multilayer structure of differet doping levels, a single measurement of the depth profile allows a calibration curve to be determined.

Tab. 1 Typical detection limits (atoms/cm^3) obtained with SIMS in GaN

dopants	detection limits	impurities	detection limits	metals	detection limits
Si	1×10^{15}	H	1×10^{17}	Cr	5×10^{14}
Mg	1×10^{15}	C	5×10^{15}	Fe	5×10^{15}
Zn	5×10^{15}	O	1×10^{16}	Mo	5×10^{15}
Cd	1×10^{16}	Cl	1×10^{15}	Ni	2×10^{16}
Se	1×10^{14}	Al	1×10^{16}	Cu	2×10^{16}
		In	1×10^{16}	Mn	5×10^{15}
				Na	1×10^{14}
				K	5×10^{13}

3.2 Matrix composition and interface quality

SIMS quantification of matrix elements is complicated because a variation in matrix composition changes the secondary ion yield and leads to a non-linear calibration curve. One technique for circumventing the matrix effect is to detect the molecular ion MCs^+ (M is the matrix element to be analyzed) under Cs^+ bombardment. Fig.2 shows the depth profiles of a GaN/InGnN/GaN quantum well structure by SIMS. Since the depth resolution (about 15 nm) is not high enough to totally resolve the well in this case, the In profile does not show a flat top in the InGaN well, which is necessary to give a single value characteristic of this layer. Consequently, the In concentration value indicated ($x_{max} = 0.084$) underestimates the real In concentration. However, a correction can be made. The areal density of In can be calculated by integrating the In profile across the quantum well. An upper limit to the In composition of $x = 0.095$ is obtained by assuming that the quantum well is a perfect delta function with a width of 34 nm (the full-width-half-maximum for the In peak). Therefore the real In concentration should be between 0.084 and 0.095.

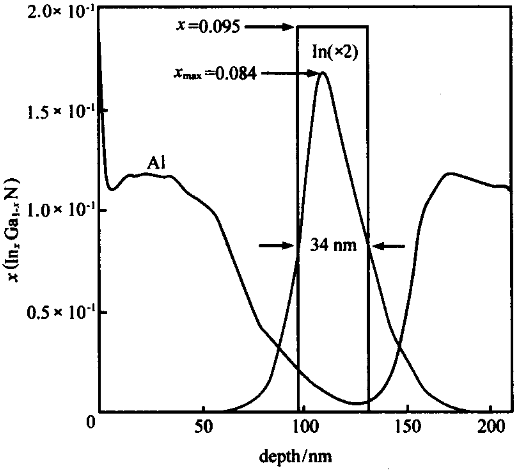

Fig.2 SIMS depth profile of a nominally 40 nm thick GaN/InGaN/GaN quantum well structure

The depth resolution in SIMS is determined by several physical processes induced by ion bombardment (cascade atomic mixing, preferential sputtering, etc.) as well as by the initial surface roughness. Generally speaking, one can achieve depth resolution no better than the initial surface roughness of the sample. For present day GaN samples, the depth resolution is mostly limited by the initial surface roughness. Even though surfaces are locally smooth on an atomic scale, the density of surface pits is great enough that the SIMS analystical area (from 10^2 to 10^3 μm^2) almost always includes some pits. These pits degrade the depth resolution to an extent which depends on their size and nature. Fig.3 shows the Al depth profiles at the interface of an AlGaN/GaN sample. In one case, some pits are visible at the surface, while in the other sample, the pits are far less numerous and invisible. Evidently, the width of the interface is broadened by the surface roughness associated with these pits. The loss of depth resolution will make the SIMS composition analysis of quantum well structures much more difficult, because secondary ion intensities characteristic of each thin layer must be measured to allow the composition to be determined accurately. Nonetheless, given the reasonably good depth resolution provided by SIMS, it can provide a straightforward means of identifying diffusion-related degradation mechanisms.

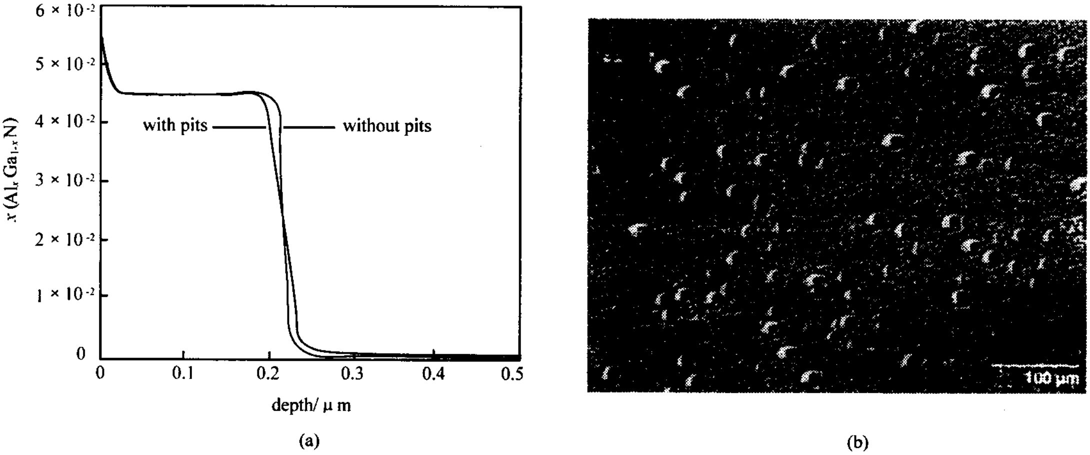

Fig.3 (a) Al depth profiles at the interface of an AlGaN/GaN sample with and without surface pits, (b) Optical micrograpth depicting the surface pits

3.3 Device analysis and reverse engineering

SIMS is a good technique to analyze finished LED devices 200 μm^2 in size. Fig.4 shows a post-SIMS measurement crater on a GaN LED device after de-encapsulation (lower left corner) and the depth profiling results consisting of both dopant and matrix ion information.

Fig.5 displays the depth profiles obtained from the LED device shown in Fig.4. SIMS is thus an excellent technique for concurrent and reverse engineering of finished devices.

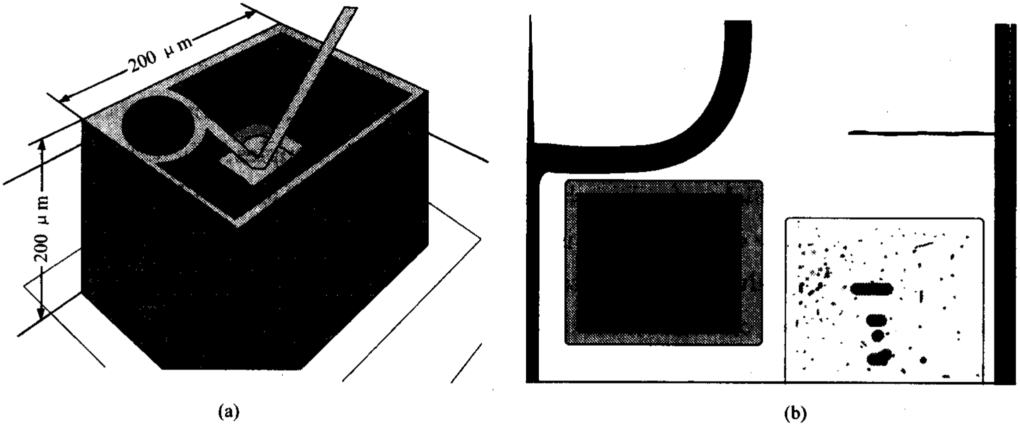

Fig.4 (a) SIMS analysis of a finished LED chip after de-encapsulation, (b) Optical micrograph depicting the post-SIMS measurement crater on the lower left side and the remnant of the setup crater on the upper right side of the device

4 Conclusion

SIMS provides the quantitative analysis for both trace and major elements (concentration ranging from 10^{15} to 10^{22} atoms/cm^3) to investigate dopant diffusion, impurity concentrations and distributions, as well as layer composition. Information on interface quality can also be obtained with the same measurement. Offering excellent sensitivity and depth resolution, SIMS is a very powerful characterization technique in both R&D and production environments.

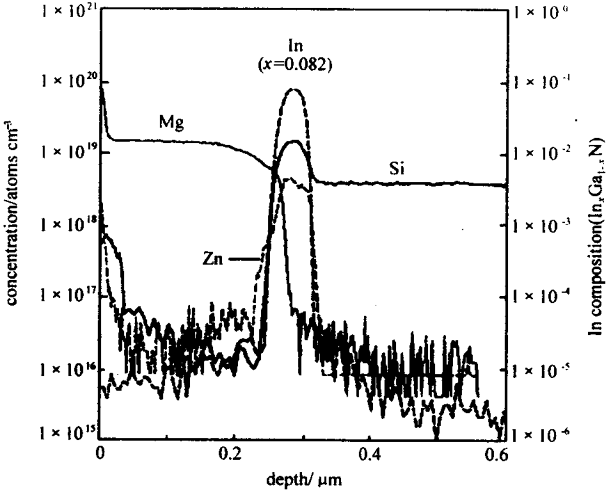

Fig.5　SIMS depth profiles of dopants compositional profile for the GaN/InGaN/GaN LED device shown in Fig.4

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