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Quantitative Secondary Ion Mass Spectrometry (SIMS) of III-V Materials

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ABSTRACT

Secondary ion mass spectrometry (SIMS) provides direct methods to characterize the chemical composition of III-V materials at major, minor and trace level concentrations as a function of layer depth. SIMS employs keV primary ions to sputter the surface and sensitive mass spectrometry techniques to mass analyze and detect sputtered secondary ions which are characteristic of the sample composition. In-depth compositional analysis of these materials by SIMS relies on a number of its unique features including: (1) keV primary ion sputtering yielding nanometer depth resolutions, (2) the use of MCs^+ detection techniques for quantifying major and minor constituents, and (3) ion implant standards for quantifying trace constituents like dopants and impurities. Nanometer depth resolution in SIMS sputtering provides accurate detection of diffusion of dopants, impurities and major constituents. MCs^+ refers to the detection of "molecular" ions of an element (M) and the Cs^+ primary beam. MCs^+ minimizes SIMS matrix effects in analysis for major and minor constituents, thus providing good quantification. This paper presents a SIMS study of $Al_xGa_{1-x}As$ structures with three different x values. MCs^+ (M=Al or Ga) data are presented for the accurate determination of major and minor components. Rutherford backscattering spectrometry (RBS) and x-ray diffraction (XRD) data were cross-correlated with the MCs^+ results. Three specimens with different x values were ion implanted with H, C, O, Mg, Si, Zn and Se to study quantification of trace levels. SIMS data acquired on a double focusing instrument (CAMECA IMS-4f) and a quadrupole instrument (PHI ADEPT 1010) are also compared. Lastly, we discuss our efforts to improve the analysis precision for p- and n-type dopants in AlGaAs which currently is $\pm$ 3% (1 sigma).

INTRODUCTION

The importance of using SIMS to support optoelectronics device manufacturing, and by implication the device development as well, is illustrated by a publication of Lucent Technologies in 2000 [1]. They used a CAMECA IMS-4f SIMS instrument to monitor dopant concentrations, layer thickness, and junction integrity for epitaxial layers of InP, InGaAs and InGaAsP doped with Zn, Fe and Si. These optoelectronics devices were laser diodes designed for high-speed transmission of digital and analog information through fiber optic media. They reported monitor data taken over a two and a half year period of manufacturing these devices.

Their SIMS measurements used the MCs^+ detection technique for quantifying major and minor constituents [2]. MCs^+ refers to the detection of "molecular" ions of an element (M) and the Cs^+ primary beam. MCs^+ minimizes SIMS matrix effects in analysis for major and minor

constituents, thus providing good quantification. They also used ion implanted reference materials to determine relative sensitivity factors (RSF's) to improve quantification [3].

EXPERIMENTAL

For the study of $Al_xGa_{1-x}As$ the following samples were used: one AlGaAs/GaAs calibration sample as quantification standard and three AlGaAs/GaAs samples for the MCs^+ experiments, and two GaAs substrate samples doped with Si and Zn, respectively. The quantification standard sample was characterized by RBS and had x_{Al} value of 0.254. The samples were ion implanted with nine species representing most common dopants and impurities (Table 1). The list of implanted ions was split into four groups to minimize possible interference.

Table 1: Ion implant parameters.

Species	Energy (keV)	Dose (at/cm^2)	Species	Energy (keV)	Dose (at/cm^2)
^{1}H	30	2E+15	^{28}Si	200	1E+14
^{9}Be	100	1E+14	^{64}Zn	300	2E+14
^{12}C	100	1E+15	^{80}Se	600	5E+13
^{18}O	100	5E+14	^{130}Te	600	5E+13
^{24}Mg	250	2E+14			

The data were taken using a CAMECA IMS-4f and a PHI ADEPT 1010 under various standard configurations with a Cs^+ primary ion beam. The H, C, O, Si, Se and Te species were measured as single charged negative ions, Be was measured as $BeAs^+$, and Mg and Zn as a molecular ion MCs^+ (M=Mg or Zn). The sputter rates were determined from profilometry measurements of the analysis craters with an Alpha step.

RBS data were taken using a 2.275 MeV He2+ ion beam with detector scattering angle of 160°. The spectra were taken with 50uC charge accumulations with the sample rotated about the incident beam to avoid ion-channeling artifacts.

DISCUSSION

The relative change in the sputter rate with composition is shown in Figure 1. The data show a linear relationship between the sputter rate and the $Al_xGa_{1-x}As$ composition for 14.5 keV Cs bombardment over the range investigated, which is in agreement with Meyer, et al. [4]. In addition, the As ion yields increase with increased Al content in Figure 2. This plot can be used for rough estimation of the Al mole fraction in AlGaAs.
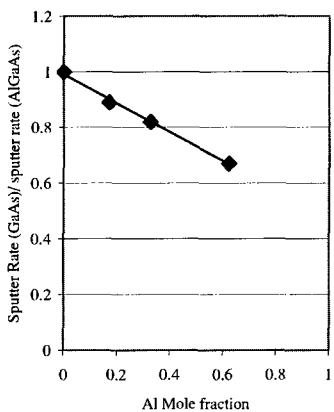
Figure 1. Relative sputter rate versus $Al_xGa_{1-x}As$ composition for 14.5 keV Cs bombardment.
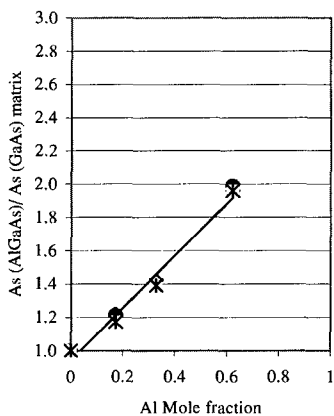
Figure 2. Relative As ion yield versus $Al_xGa_{1-x}As$ composition for 14.5 keV Cs bombardment.

Measured ion counts are converted to concentrations by Relative Sensitivity Factors (RSF's). These are highly specific for each species and matrix as illustrated in Figure 3. Relative RSF's are plotted versus $Al_xGa_{1-x}As$ composition. The change in relative RSF reflects changes in both the ion yield for the species and ion yield for the matrix species. For species with a high ionization potential like Se and Te, the ion yield is constant in the various AlGaAs matrixes. Therefore, the relative RSF reflects mainly the change in ion yield for the As⁻ matrix species. For species where the change in the ion yield is proportional to ion yield change of the matrix the relative RSF remains constant [5]. The MCs^+ detection technique has much reduced ion yield changes as is illustrated by the small change of relative RSF's for both Mg and Zn.
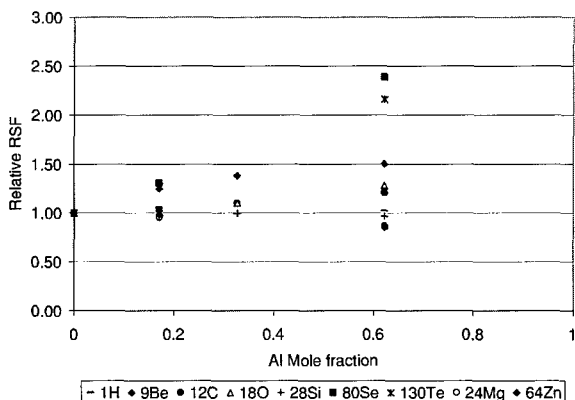
Figure 3. Relative RSF for implanted species as function of $Al_xGa_{1-x}As$ composition

Table 2 illustrates a high precision study for SIMS data acquired from a Se doped AlGaAs

Table 2: High precision SIMS analysis of Se dopant in AlGaAs.

	Se in AlGaAs (at/cm^3)		
SIMS Measurement	Reference Standard	Sample A	Sample B
1	7.54E+17	1.22E+18	1.38E+18
2	7.66E+17	1.21E+18	1.34E+18
3	7.42E+17	1.19E+18	1.35E+18
4	7.44E+17	1.20E+18	1.36E+18
5	7.30E+17		
6	7.66E+17		
7	7.45E+17		
8	7.33E+17		
9	7.64E+17		
Average	7.49E+17	1.21E+18	1.36E+18
% Standard Deviation	1.85%	1.29%	1.71%

structure. The reference standard provided the quantification for this work in which repeated measurements of this reference determined the analytical precision, in this case 1.85% (1 sigma). Samples A and B are analytical samples which yield the tabulated measurement and average concentration values. The variance of these latter values fall within the precision of the standard measurements insuring that instrumental variations are not distorting the measured concentrations of the Se dose. In addition, the high level of measurement precision demonstrates that SIMS is capable of detecting small differences in the Se concentration.

Optimal SIMS materials analysis requires statistical process control (SPC) procedures to insure the quality of the data. In our laboratory, SPC procedures utilize both calibration and control standards in which the calibration standard is used to quantify the sample while the control standards are used to evaluate reproducibility. Figure 4 illustrates a recent SPC plot for dopant concentration of p doped AlGaAs, over the past three months. This plot shows an approximate ±3% (1 sigma) precision over this time frame.
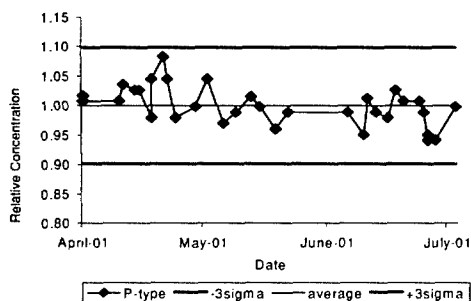

Figure 4. Relative dopant concentration of p type AlGaAs.

Table 3: Mole fraction $Al_xGa_{(1-x)}As$

	A	B	C
	0.168	0.318	0.622
SIMS	0.173	0.332	0.621
	0.171	0.330	0.624
Avg	0.171	0.327	0.622
Std Dev	0.3%	0.8%	0.2%
RBS	0.16	0.28	0.60
XRD	0.149	0.307	0.615

A representative SIMS compositional analysis of $Al_xGa_{1-x}As$ using a Cs^+ primary ion beam and negative secondary ion detection for three specimens having different x values is illustrated in Table 3. The SIMS data is compared to data acquired from Rutherford Backscattering Spectroscopy (RBS) and x-ray diffraction (XRD). The SIMS precision is less than 1% and the measured values correspond well with the accurate data obtained by RBS and XRD. The major advantage of the SIMS technique in this type of application is its ability to perform compositional analysis of layered structures.

Clear examples of the power of SIMS depth profiling are illustrated in Figures 5 and 6 which plot the results of a high resolution depth profile of a InP/GaAsSb/InP DHBT device using a Cs^+ primary ion beam and negative secondary ion detection. The sample had a planar structure and the relative positions and secondary ion intensities accurately map the composition of the device to a depth of 800 nanometers. High depth resolution allows dopant uniformity through thin layers to be evaluated and the location of contaminants (often at interfaces) to be determined. Figure 4 is a blow up of the central region of this depth profile illustrating the distribution of a 50nm wide C dopant from which quantitative data can be obtained.
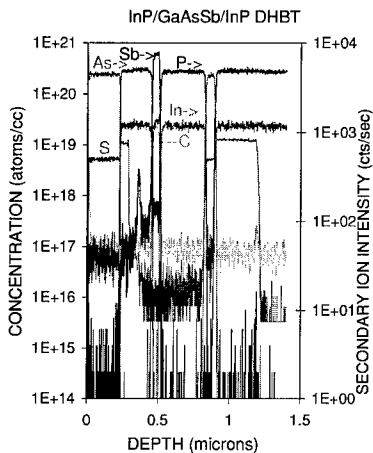
Figure 5. Sulfur and carbon profiles were collected concurrently with layer marker profiles using a 2 keV Cs primary ion beam.
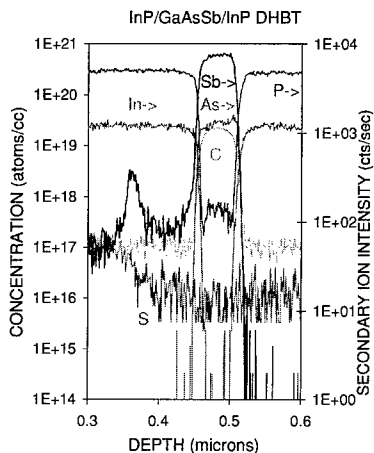
Figure 6. Detail of 50 nm C doped layer

CONCLUSIONS

State-of-the-art secondary ion mass spectrometry techniques and methods can address many of the dopant, impurity and compositional analysis issues facing the development of innovative III-V materials. The high detection sensitivity and in-depth resolution of SIMS make it one of the most powerful materials analysis techniques. The implementation of SPC protocols along with the analysis of standard materials insure a high degree of reliability in SIMS data in process monitoring applications.

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