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ULTRA-LOW ENERGY SIMS DEPTH PROFILE ANALYSIS OF MOVPE GROWN InAlGaAs/AlGaAs/GaAs NANOSTRUCTURES

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Abstract. Ultra-low energy secondary ion mass spectrometry (SIMS) depth profile analysis was performed on metal organic vapour phase epitaxy (MOVPE) grown structures. The layered systems are multi quantum well (MQW) structures composed of InAlGaAs, AlGaAs and GaAs. The structures typically consisted of GaAs substrate, 150 - 500 nm GaAs buffer, MQW region and 50 - 70 nm GaAs cap layer. The MQW is a 3-layer period superlattice made of 4.5–9 nm thick $In_xAl_yGa_{1-x-y}As$ layers and 30 nm thick GaAs or $Al_zGa_{1-z}As$ layers.

SIMS analyses performed with the use of 880 eV Ar^+ ion beam give depth profile resolution (16%-84%) of ~3 nm measured at GaAs/InAlGaAs interface of MQW structure. Comparison of SIMS data with high resolution X-ray diffraction (HRXRD) allows to measure thickness of particular layers in the studied structures as well as indium molar fraction in quaternary compound InAlGaAs.

1. INTRODUCTION

Characterisation of layered nanostructures requires the use of sensitive and precise techniques in depth profile analysis. Ion sputtering is one of the methods, which allows removal of atomic layers of the structure. Recently sputtering with ultra-low energy (below 1 keV) ion beams is used in order to reduce ion beam mixing effects [1,2]. Here we show the results of 880 eV argon ion beam sputtering applied in secondary ion mass spectrometry (SIMS) of metal organic vapour phase epitaxy (MOVPE) grown structures. The results are compared with X-ray diffraction analysis performed on the same heterostructures.

2. EXPERIMENTAL

The analysed structures were obtained by AP MOVPE system using AIX 200 R&D Aixtron reactor. The growth temperature (T_g) was in the range of 650 ÷ 670 °C. The H_2 flow rate through TMIn bubbler ($V_{H2/TMIn}$) was varied from 35 to 75 sccm. The H_2 flow rate through TMAl bubbler was varied from 1 to 4 sccm. The H_2 flow rate through TMGa bubbler was 1.5 sccm. The other parameters were the same as described in ref. [3, 4]. The test structures consisted of three types of MQW region were grown on semi-insulated GaAs (100) substrate (Table 1).

Technological parameters of the MOVPE growth process were changed in order to obtain different compositions of $In_xAl_yGa_{1-x-y}As$ and $Al_zGa_{1-z}As$ layers. For example the highest In content (x) in the $In_xAl_yGa_{1-x-y}As$ layer was obtained for the structure Al-05, prepared in T_g = 650 °C (x = 28.5%). In the higher temperature (T_g = 670 °C) and with the same H_2 flow rate through the TMIn bubbler, the In content in the sample was about 30% lower (structure Al-06, x = 21%).

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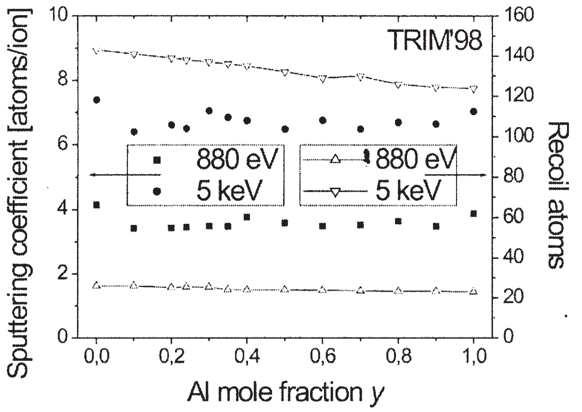

Fig. 1. Sputtering yield and number of recoils per incident ion during bombardment of $Al_yGa_{1-y}As$ matrix with 880 eV and 5 keV Ar^+ ions shown with respect to the Al mole fraction (y). Presented results are obtained from Monte Carlo TRIM'98 simulation.

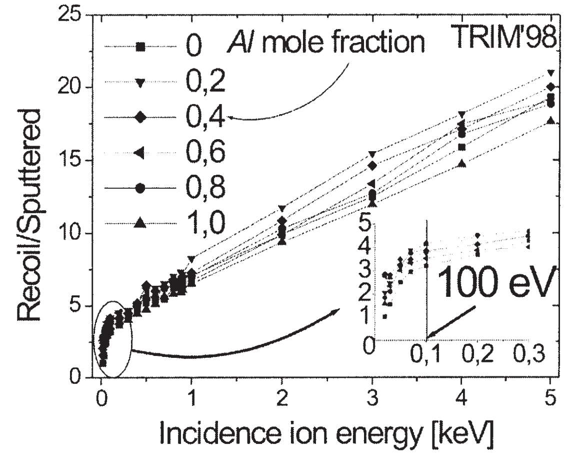

Fig. 2. Number of recoil atoms per one sputtered atom of $Al_yGa_{1-y}As$ matrix measured versus incidence beam energy (TRIM'98 simulation of Ar^+ bombardment).

SIMS analysis of the above mentioned structures was performed on *SAJW-05* apparatus equipped with 06-350E Physical Electronics ion gun and QMA-410 Balzers 16 mm quadrupole analyser. Argon ion beam sputtering was used with the two energies 5 keV and 880 eV. The beam (100 μm diameter) was digitally rastered over 1 mm×1.6 mm area. The resulted ion eroded craters were measured *ex-situ* with the use of Tencor alpha-step 100 profilometer in order to calibrate sputtering rate during depth profile analysis. Sampling distance in SIMS measurements was: 0.2–0.6 nm for 880 eV energy of ion bombardment and 6–10 nm for 5 keV energy. Depth resolution Δz (16% - 84%) measured at a rising slope of In^+ secondary ion current, was 3.5–7.5 nm for 880 eV and 8–15 nm for 5 keV. Sputtering rate during depth profile analysis was 12–16 nm/min for 5 keV. Much lower values of sputtering rate (0.5–1 nm/min) have been obtained for 880 eV bombardment.

Table 1. Three types of MQW structures grown by MOVPE and analysed by SIMS and HRXRD.

Sample #	Structure
Al-02, Al-05	3×GaAs(well) / $In_xAl_yGa_{1-x-y}As$ (barrier)
Al-06, Al-07	3×$In_xAl_yGa_{1-x-y}As$(well) / $Al_zGa_{1-z}As$ (barrier) with GaAs cap layer
Al-08, Al-09	3×$In_xAl_yGa_{1-x-y}As$(well) / $Al_zGa_{1-z}As$ (barrier) without GaAs cap layer

Monte Carlo simulations of sputtering process and ion mixing effects were done using TRIM'98 code for the matrices $Al_yGa_{1-y}As$ using several input parameters like density, ranging from 5.316 g/cm^3 for $y = 0$ to 3.729 g/cm^3 for $y = 1$, displacement energy 15 eV, binding energy 2 eV and average surface binding energy 2.48 eV. Each simulation was performed for 1000 argon ions in the range of 15 eV to 5 keV energy and angle of incidence 45°.

X-ray diffraction was performed on high resolution Philips Materials Research Diffractometer (MRD) using a four-crystal Bartels monochromator and Bonse/Heart analyser. For simulations we used PC-HRS program supported by Philips, and the results were compared with experimental data. The applied procedure allowed to suggest stoichiometry coefficients as well as layer thickness of the particular structures.

3. RESULTS

Prior to depth profile analyses, Monte Carlo simulations of the sputtering process were performed. Sputtering yields and number of recoil atoms were monitored for different Al mole fractions in $Al_yGa_{1-y}As$ matrices – Fig. 1. The results show that both sputtering yield and number of recoil atoms are al-

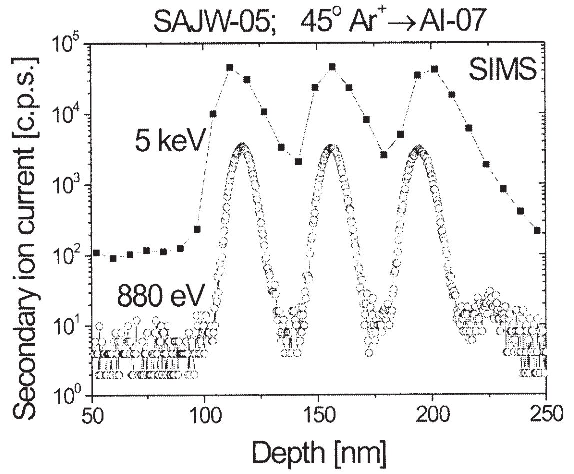

Fig. 3. $^{115}\mathrm{In}^+$ SIMS depth profile analysis performed with the use of 5 keV and 880 eV Ar^+ ion beams for Al-07 MQW structure.

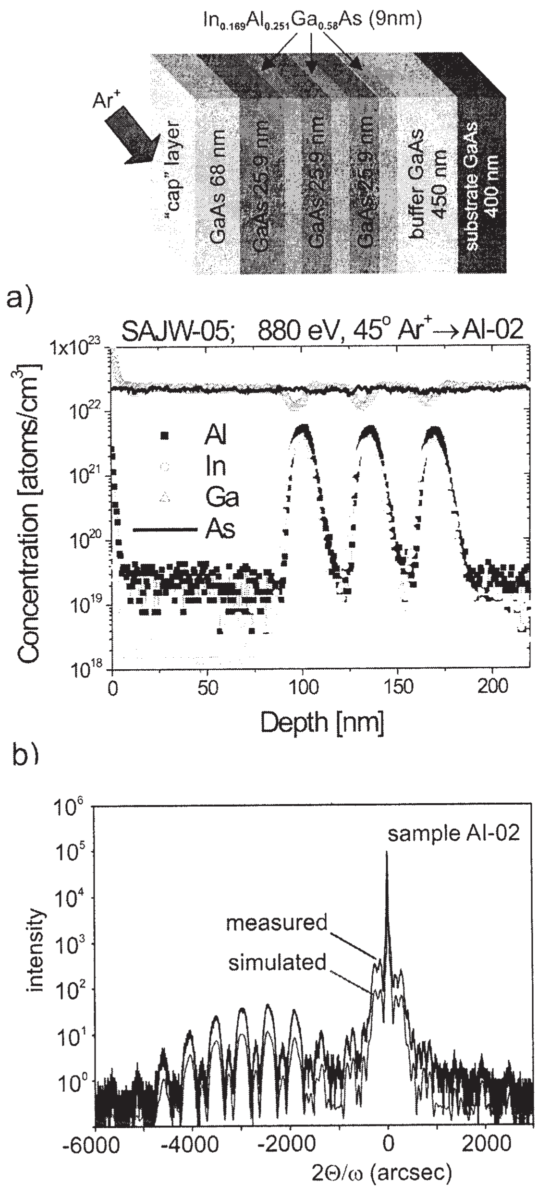

Fig. 4. Results of SIMS depth profile (a) and HRXRD analyses (b) of MQW GaAs/InAlGaAs MOVPE grown structure (Al-02). The schematic of this structure is shown above. Atomic concentrations at SIMS profiles have been calibrated basing on XRD data. SIMS depth scale have been calibrated basing on stylus profilometry measurements.

most not dependent on the Al mole fraction in this matrix. Number of recoil atoms, measured with respect to the number of sputtered atom flux may represent the effective mixing occurring during SIMS depth profiling. The beam energy dependence of this ratio is shown at Fig. 2. We see that when the primary ion beam energy is lower, the number of recoils per one sputtered atom is reduced. The most favourable energies recommended for sputtering of the $\mathrm{Al}_y\mathrm{Ga}_{1-y}\mathrm{As}$ matrix would be those below 100 eV, however this requirement for ion optical system is very difficult to fulfil experimentally. Also sputtering rate as well as secondary ion signals would be very low for such energies. Instead, the lowest energy applied in this study was 880 eV, in this case one out-coming sputtered atom leaves about six recoils in the near-surface altered layer. In comparison, ion bombardment with 5 keV energy causes about 20 recoils within the altered layer per one sputtered atom. Experimental data of SIMS depth profiles obtained with the use of 5 keV and 880 eV ion bombardment show significant difference in depth resolution – Fig. 3.

All MOVPE prepared structures were analysed by two different analytical methods SIMS and HRXRD. SIMS depth profiles were performed with both 5 keV and 880 eV ion beams. SIMS allowed to determine the structure by direct analysis of main components of the structures – the chosen secondary ions were $^{27}\mathrm{Al}^+$, $^{69}\mathrm{Ga}^+$, $^{75}\mathrm{As}^+$ and $^{115}\mathrm{In}^+$. SIMS depth profiles of the two chosen MQW structures and one reference structure are shown in Figs. 4 - 6. In comparison also HRXRD results of the structures are shown.

Fig. 4 presents the results obtained for Al-02 structure composed of the three InAlGaAs layers separated by GaAs layers. Secondary ion currents of indium and aluminium indicate the presence of three separate InAlGaAs layers. Conversion of the

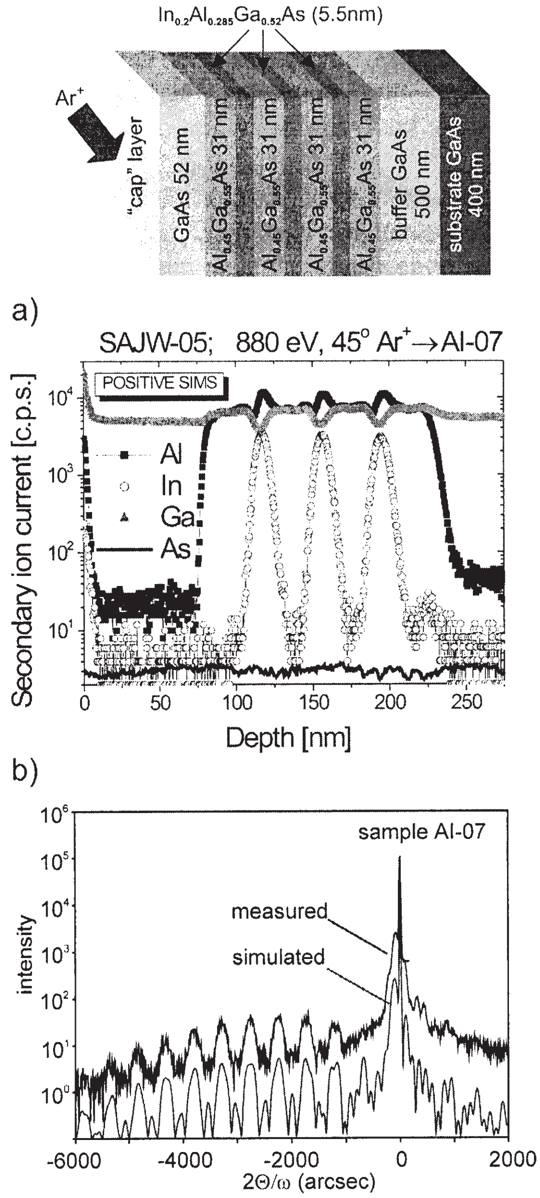

Fig. 5. Results of SIMS depth profile (a) and HRXRD analyses (b) of MQW AlGaAs/InAlGaAs MOVPE grown structure (Al-07). The schematic of this structure is shown above. Atomic concentration calibration for SIMS analysis has not been performed due to the strong matrix effect present at the GaAs/AlGaAs and AlGaAs/InAlGaAs interfaces.

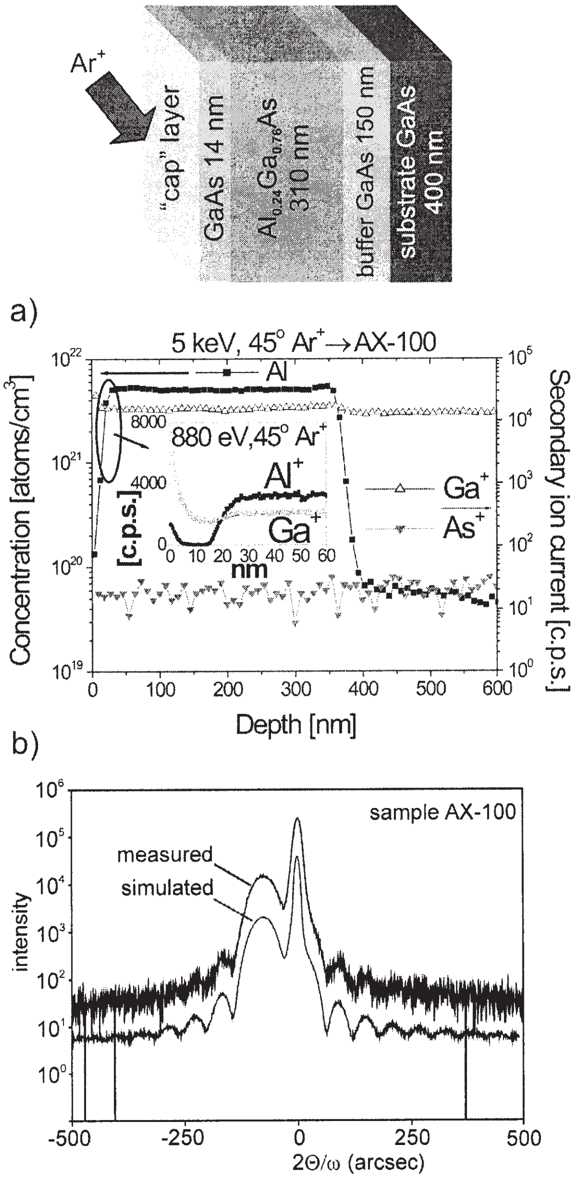

Fig. 6. Results of SIMS depth profile (a) and HRXRD analyses (b) of AlGaAs/GaAs MOVPE grown test structure (AX-100). The schematic of this structure is shown above. SIMS profiles have been performed using both 5 keV and 880 eV ion beams. Atomic concentration calibration has been performed only for aluminium.

ion currents to concentration was done basing on the HRXRD measurements of this structure. Fig. 5 shows results of the other MQW structure Al-07, composed also of three InAlGaAs layers. In this case the wells are separated by AlGaAs barriers. The graph shows original SIMS data without current to concentration conversion. The conversion can not be done due to the strong matrix effect of Al^+ and Ga^+ ion currents emitted from AlGaAs and GaAs matrices. This effect causes in particular that Ga^+ ion current emitted from the AlGaAs matrix is higher than the emitted from GaAs while in fact the Ga concentration is higher in GaAs.

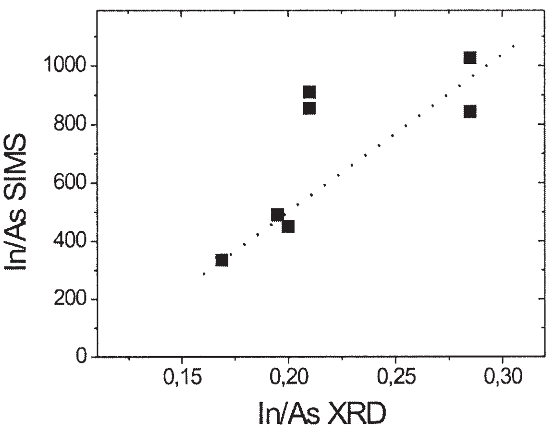

Fig. 7. Comparison of SIMS and XRD measurements of quaternary InAlGaAs matrix. Secondary ion current ratios measured with SIMS has been compared with concentration ratios of In/Al measured by XRD.

Another structures have been grown mainly for calibration purposes. Fig. 6 shows the results of one of such structures AX-100. In this case SIMS depth profile shows Ga and As ion signals (right axis) and Al concentration (left axis). According to *X*-ray analysis, this structure is composed of 150 nm GaAs buffer and relatively thick (310 nm) single AlGaAs layer covered by 14 nm GaAs cap layer. Depth calibration of SIMS results compared with crater depth measurements performed with stylus profilometer, however, suggests 360 nm thick AlGaAs layer with 20 nm GaAs cap layer. Comparison of layer thickness measurements of the whole set of structures analysed by XRD and SIMS is shown in Table 2. Full width at half maximum (FWHM) data of SIMS signals of In and Al, representing InAlGaAs layers, are higher than XRD data by 1 nm to 3.5 nm, mainly due to ion beam mixing present during SIMS sputtering. The SIMS data of AlGaAs layer thickness are equal or higher than XRD data.

Beside the layer thickness calibration, SIMS and XRD data allowed also to suggest molar compositions of analysed layers. Indium concentration in quaternary InAlGaAs layers obtained from X-ray analysis was compared with the SIMS data. Fig. 7 presents indium concentrations shown in relation to arsenic concentration for different matrices measured with XRD and SIMS (XRD value of x between 0.169 and 0.285). Four of five structures fall approximately in linear dependence, while one structure (XRD value of $x = 0.210$) stays apart of this dependence. Also ternary $Al_yGa_{1-y}As$ layer composition was compared for XRD and SIMS. Two structures AX-100 and AX-111 with XRD y values of 0.24 and 0.685 respectively and relatively thick ternary layers were depth profiled by SIMS. The data obtained from the other thin ternary layers of QW structures show that measured Al/Ga SIMS signal ratio is independent of composition described by XRD analysis. Earlier study [3], performed on $Al_yGa_{1-y}As$ matrices with much higher energy of Ar^+ bombardment (15 keV), shows linear dependence of Al/Ga and Al/As SIMS ratios of concentration up to $y = 0.5$ but no such relation of Ga/As ratio. In order to describe these relations for ultra-low energy bombardment, it is necessary to prepare several other reference samples of $0.1 < y < 0.6$.

Table 2. Layer thickness measurements. Results are obtained from SIMS depth profiles calibrated with stylus profilometry and XRD characterisation.

Sample #	InAlGaAs thickness [nm]		AlGaAs layer thickness [nm]		GaAs layer (well) thickness [nm]	
	SIMS	XRD	SIMS	XRD	SIMS	XRD
Al-02	10	9	–	–	25	25.9
Al-05	12	8.5	–	–	26	27
Al-06	12	8.5	39	30	–	–
Al-07	8.5	5.5	31	31	–	–
Al-08	8	6.2	30	30	–	–
Al-09	7	4.5	31	29.7	–	–
AX-100	–	–	345	310	–	–
AX-111	–	–	525	465	–	–

4. CONCLUSIONS

The presented results show the possibilities of ultra-low energy SIMS in analysis of InAlGaAs/AlGaAs heterostructures. Depth profile resolution is about two times better than in conventional 5 keV SIMS. However, reduction of energy down to 880 eV reduces also sputtering rate and measured signals. SIMS layer thickness calibration gives total values in good accordance with XRD data.

Both ultra-low energy SIMS and HRXRD analyses indicate high quality of MQW region interfaces. The applied technological parameters enable us to prepare the high quality of InAlGaAs/AlGaAs heterostructures for optoelectronic application.

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