

REVIEW PAPER

Characterisation of Semiconductor Materials/Device Structures using SIMS

Anuradha Dhaul, S.K. Sharma, R.K. Sharma, and A.K. Kapoor

Solid State Physics Laboratory, Lucknow Road, Delhi-110 054

ABSTRACT

Secondary ion mass spectrometry (SIMS) is an analytical technique that can be used to characterise the surface and near-surface region of solids. The instrument operation and data analysis have been discussed to obtain meaningful results. The paper discusses the technique of sequential sputtering to elucidate the thickness of individual layers in a multilayer structure. The application of the technique for failure analysis, standard generation and interface studies have been discussed in detail taking examples of multilayer structures of compound semiconductors being developed at SSPL.

Keywords: SIMS, ion implantation, heterostructures, depth profiling, secondary ion mass, spectrometry

1. INTRODUCTION

Secondary Ion Mass Spectrometry (SIMS) is an analytical technique that can be used to characterise the surface and near-surface (from a few nanometers up to a few micrometer) region of solids^{1,2}. SIMS is used to evaluate dopant profiling and trace contamination^{3,4} (up to ppm-ppb levels) of surfaces, thin films, thick films, multilayer structures, and interfaces. It is used to measure relative levels of incorporated impurities or component elements as a function of processing parameters⁵. Samples must be compatible with the ultra-high vacuum (approximately 10^{-9} Torr) of the SIMS analytical chamber⁶. This is required to prevent re-absorption of sputtered species and permanent gases onto freshly ion milled sample surfaces.

A few case studies on application of SIMS techniques for: (a) reliability studies on ion implantation process, (b) depth profiling of dopant/impurity concentration, (c) identification and estimation of various layers and interfaces in multilayer structures, (d) interface examination⁷, and (e) process development/Failure analysis⁸, are presented in this review.

The work reported has been carried out at SSPL using a CAMECA IMS 4F SIMS machine.

2. METHODOLOGY

The specimen under study was bombarded by a beam of energetic particles (primary ions usually O_2^+ , Cs^+ , O^-) in the energy range of 0.5 keV to 20 keV. The primary ions, in the energy range used in this process, when impinging into the solid, transfer kinetic energy to the target atoms via elastic collisions. A target atom set in motion by such an elastic collision, in turn, transfers a part of its energy to another target atom. In this way, a collision cascade⁹ is generated, causing a large number of target atoms to be set in motion (Fig.1). During this cascade generation, some of the target atoms in the near-surface region

(~ 10 Å in depth) receive enough outward directed momentum, and hence sufficient energy to overcome the surface potential barrier and leave the target¹⁰. A portion of the sputtered atoms undergoes a charge exchange in the near-surface environment, resulting in their conversion to positive or negative secondary ions. These secondary ions are then extracted via an electrical potential and subsequently analysed by a mass spectrometer. Detection of the secondary ions is by an electron multiplier, Faraday cup, or ion-sensitive image amplifier for imaging. Figure 2 displays a typical instrumental arrangement of the primary ion source and the mass analyser. Secondary ions carry information about the composition of the specimen surface. As sputtering proceeds, layer-by-layer information for deeper layers is acquired in the form of a depth profile. The precision with which the atomic layers of the specimen are removed by ion beam sputtering combined with the very high mass resolution, makes SIMS a very useful tool to study compositional depth profiles.

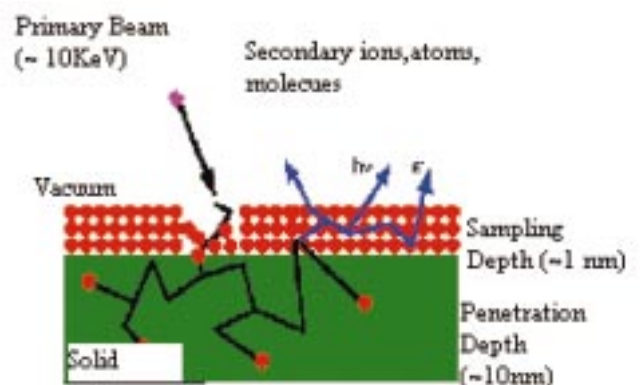


Figure 1. Schematic of sputtering process.

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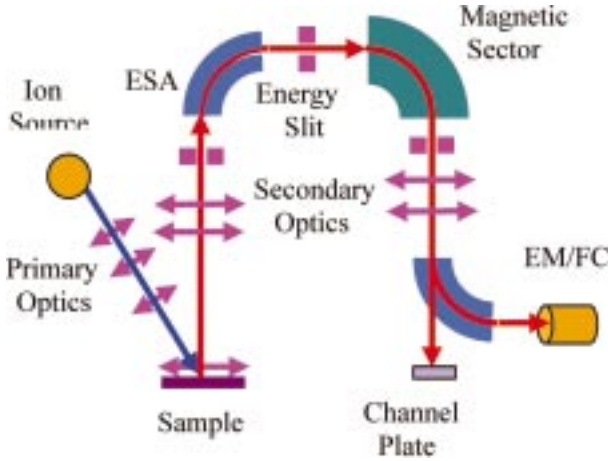


Figure 2. Layout diagram of magnetic sector.

3. QUANTIFICATION

For a meaningful interpretation of the SIMS data, the information obtained in the form of secondary ion yield (counts) versus sputtering time needs to be translated to concentration (atoms/cm³) versus depth (nm). The two factors called the sputter rate and relative sensitivity factor are used for this purpose. The samples are categorised as (i) bulk structures and (ii) single layer/multilayer structures.

3.1 Bulk Samples

3.1.1 Depth measurement

For samples with uniform composition, well-defined shallow craters, formed by ion beam bombardment, are measured using a stylus profilometer, Dektak, and Sputter Rate (SR) determined as follows:

$$SR = \text{crater depth } (d) / \text{sputter time } (t) \quad (1)$$

By calibrating the sputter rate under specific experimental conditions, the time scale is converted to the sputtered depth. These SR values can be used for depth determination of deep craters formed under identical conditions for similar matrices.

3.1.2 Concentration (atoms/cm³)

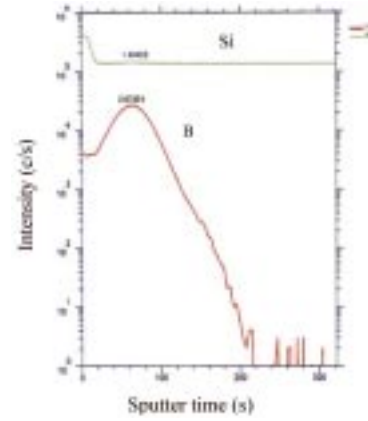
Relative sensitivity factor (RSF) is a conversion factor from secondary ion yield (counts) of the dopants/impurities to the atom density (atoms/cm³). It is defined with reference to one of the matrix elements as follows.

The concentration of any species A relates to the secondary ion yields as

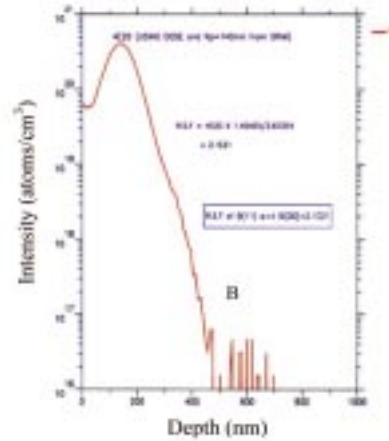
$$n_A = (I_A^\pm / I_M^\pm) \times RSF_{AM} \quad (2)$$

where I_A^\pm and I_M^\pm are the positive or negative secondary ion yield (counts) of species A and matrix ion M respectively. The relative sensitivity factor (RSF) of an element in a particular matrix can be determined from an ion implanted calibration sample^{11,12} as described below:

Figure 3(a) shows the depth profile of a calibration standard of boron implanted in silicon with known dose and energy. The standard was procured from Charles Evans and Associates, who are analysts of international repute.



(a)



(b)

Figure 3. (a) SIMS depth profile of a standard B in Si implant and (b) Quantified depth profile of B corresponding to data of (a).

From the known dose, the peak concentration, n_A , can be theoretically determined. Using the known dose and secondary ion yield of boron and silicon elements, RSF value is generated for boron in silicon matrix by substituting in Eqn. (2). The RSF values determined are used as standards for unique impurity-matrix combinations.

The projected range, corresponding to the implant energy, determines the peak position in nm. Fig. 3(b) shows the quantified depth profile corresponding to the data of Fig. 3(a). The complete profile is extrapolated with the help of software.

Generation of Standards

(i) Standards are usually generated using Ion implantation data:

Ion implanted samples can serve as a standard for SIMS calibration. Isotope (more than one also) of the required element can be implanted provided the fluences of all isotopes can be determined. The precision (error) in the dose concentration can be determined by carrying out round robin depth profiling on the calibration standards.

(ii) RSF bank is available for some matrix-element combinations¹³.

3.2 Single Layer/ Multilayer Structures

3.2.1 Depth Measurement

Since the sputter rate is a function of composition, it will vary within the depth of the sample comprising of the same matrix but with varying composition (x value); e.g. $Ga_xAl_{1-x}As$, $Hg_{1-x}Cd_xTe$ (MCT), etc. In case of MCT, a linear relationship has been reported¹⁴ for SR vs x-value. Thus, the variation in SR due to varying composition can be taken into account through tedious mathematical modelling if all the material parameters are known. For structures comprising of layers having different constituents in each of the layers, a sharp change in the secondary ion yield can be observed on depth profiling through the different layers. Such structures are being referred to as multilayer structures. A straightforward approach, as described below, referred to as Sequential Sputtering¹⁵ has been evolved for thickness estimation in multilayer structures.

For quantifying the time scale to obtain depth in the case of multilayer structures, the following steps were carried out:

- Possible interfaces were located from a complete depth profile.
- Sequential profiling up to individual interfaces was carried out.
- Crater depth measured for each crater using Dektak surface profilometer.
- Complete depth profile for the structure was reconstructed.

Figure 4 shows the depth profiling on the initial few layers of a $GaAs$ HEMT structure where sequential sputtering is depicted for thickness estimation of the top $GaAs$ layer, followed by $GaAlAs$ layer and then the In $GaAs$ layer. The sharp rise in the Al signal, (Fig. 4(a)), indicates the start of $GaAlAs$ layer and end of $GaAs$ layer. The crater measured for this, using DEKTAK, corresponds to the thickness of the top $GaAs$ layer. Figure 4(b) shows the region corresponding to a constant Al signal and the profiling is abruptly stopped as the In signal rises and Al signal falls. The crater measured corresponds to the top two layers of the structure, namely $GaAs$ and $GaAlAs$. The next crater is formed, (Fig. 4(c)), till the fall of the In signal; resulting in a crater comprising

of $GaAs$, $GaAlAs$ and $GaInAs$ regions.

This process can be continued up to the end of the entire structure for thickness estimation of the individual layers in a multilayer structure.

1.2.2 Concentration

The quantification of multilayers is not so straightforward and yields semi-quantitative results. The secondary ion yield of a particular element strongly depends on its chemical environment. This causes variations in the secondary ion yield of an element over several orders of magnitude from one matrix to another. Therefore, SIMS not only has a wide variation in secondary ion yield between different elements, it also shows strong variations in the secondary ion yield from the same element in different matrices. This is called the matrix effect¹⁶ and is the most serious SIMS artifact.

To analyse the distribution of dopants/impurities in each of the layers, individual RSF values can be applied for each element-matrix combination. The problem, however, occurs at the interfaces where there is a lot of intermixing and the matrix cannot be defined uniquely. The use of RSF for concentration determination is strictly valid only at low level of impurities ($<10^{19}cm^{-3}$) and may not be appropriate for a matrix component. For quantifying the intensity data, for matrix elements ($>10^{19}cm^{-3}$), layers with known composition are taken as reference for self-normalisation. An example of this is presented in Section (e) where the intermixing of metallic multilayer structures was investigated. The normalised counts correspond to a value of 1 in the region where 100 % of that metal is present. This again results in semi-quantitative information, as the secondary ion yield of the metal under consideration will be strongly influenced due to the matrix effect.

Following are a few case studies indicating the usefulness of SIMS as a characterisation tool for semiconductor materials and device structures.

4. CASE STUDIES INDICATING USEFUL

4.1 Reliability Studies on Ion Implantation Process

Dopants may be introduced into semiconductor materials for device formation by ion implantation, diffusion or introduced during the growth of semiconductor epilayers. The validation of ion implantation parameters, namely the implant dose and energy, for concentration and junction depth determination, is an important material characterisation step. This requires the following tests to be carried out using SIMS.

- Estimation of ion energy and dose with unknown implantation parameters.
- Assessment of lateral/ run-to-run uniformity of implantation profiles.

A typical case of B implanted in Si with unknown dose and energy is presented for two samples prepared in-house by the Ion implantation Group; standard sample of B implanted in Si, with known dose and energy, was available from Charles Evans. The following procedure was adopted:

- RSF of B wrt. Si determined using standard sample.
- Depth profiling on samples under study carried out

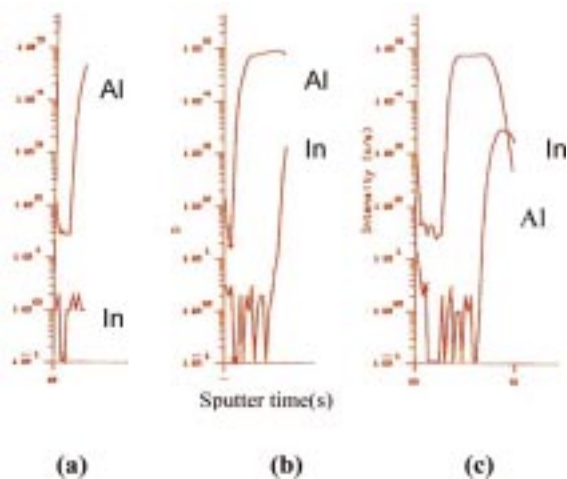


Figure 4. Sequential profiling up to individual interfaces.

and data quantified using RSF values for conc. determination and thickness measurement using DEKTAK.

- (iii) Peak implant concentration (N_{max}) and position (R_p) determined from the SIMS profile.

Figure 5 shows the depth profiles for B in Si with different dose and energy corresponding to the two samples (S1 and S2). The values obtained from SIMS profile for S1 are R_p at 180 nm and N_{max} 4.0×10^{19} atoms/cm³; for S2 are R_p at 410 nm and N_{max} 6.1×10^{18} atoms/cm³.

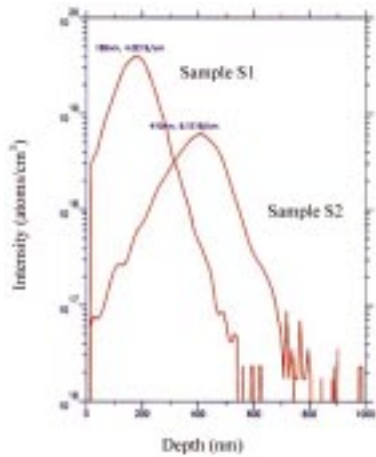


Figure 5. Depth profiles for two ion implanted samples

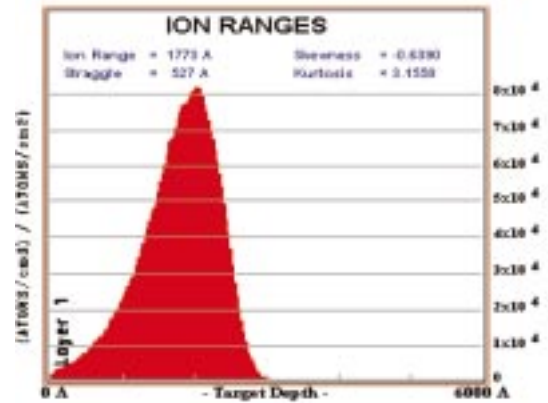
- (iv) Implantation energy corresponding to this peak position is determined by matching with the Monte–Carlo simulation program SRIM 2000. The nearest values obtained using SRIM 2000 (which is a standard code available as freeware on Internet) correspond to implantation energy of 50 keV for S1 and 130 keV for S2. Figure 6(a) and (b) show the SRIM ion range distribution corresponding to these values with R_p at 178 nm and 425 nm respectively.
- (v) Substituting for N_{max} from Fig. 5 and R_p from Fig. 6 in the approximate theoretical relation $N_{max} = Dose / 0.4 \Delta R_p$, the dose for the two samples is determined.

Table 1 compares the values of ion energy and dose determined by this method with the intended values. The close agreement between the two validates the ion-implanted parameters.

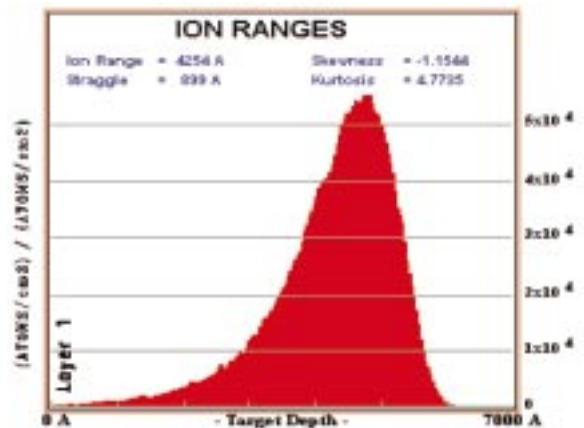
Samples are also examined for the purpose of examining the lateral uniformity across the surface of the samples. A test sample of 150 keV B⁺ implant in Si with a dose of 5×10^{14} atoms/cm² is presented for the purpose of a case study. Figure 7 shows the SIMS depth profiles taken at three different points A, B and C nearly 1 cm apart. Extremely

Table 1. Comparison of estimated and targeted values of ion energy and dose

Sample ID	Ion range (nm)		Ion Energy (KeV)		Dose (cm ⁻²)	
	SIMS	SRIM	Estimated	Targeted	Estimated	Targeted
S1	180	178.4	50	50	5.0×10^{14}	5×10^{14}
S2	410	424.3	130	130	1.1×10^{14}	1×10^{14}



(a)



(b)

Figure 6. (a) SRIM simulation for B in Si at 50 keV and (b) SRIM simulation for B in Si at 130 keV.

good lateral uniformity is observed in the depth profiles. Table 2 compares the salient values for the three distributions and the simulated profile (Fig. 8) under similar conditions.

Figure 9 shows the extremely good agreement in the depth profiles obtained using dose (implantation data) and RSF (SIMS data). Thus, an ion implanter yielding correct dose and energy values can also be used for generating

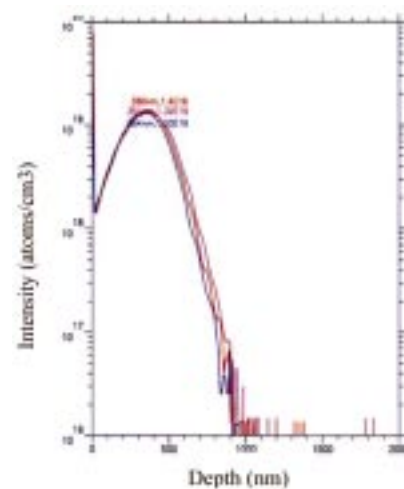


Figure 7. SIMS depth profiles taken at three different points.

Table 2. Comparison of the salient values for the three distributions and the simulated profile

	R_p	N_{max} (cm^{-3})
Point – A	0.36 μm	1.4×10^{19}
Point – B	0.35 μm	1.3×10^{19}
Point – C	0.36 μm	1.3×10^{19}
Simulation	0.35 μm	1.3×10^{19}

implantation profiles of dopants in matrices with known dose and energy, which can in turn be used as standards for the purpose of SIMS quantification (RSF generation). SIMS and ion implantation go hand in glove. SIMS is the most suited technique for validation of ion implantation parameters and Ion implantation is the most suited technique for generation of standards required for SIMS quantification.

4.2 Depth Profiling of Dopant/Impurity Concentration

A representative case of impurity analysis in Mercury Cadmium Telluride (MCT) epitaxial layers grown on Cadmium



Figure 8. Simulated profile using SUPREME software.

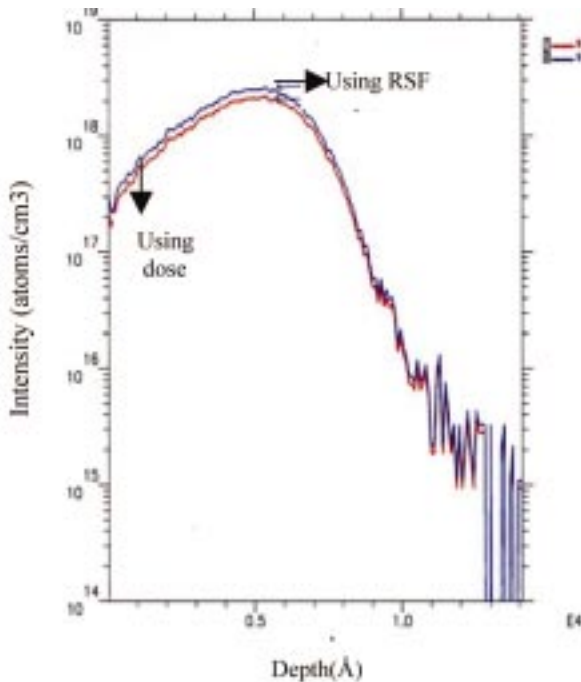


Figure 9. Depth profiles generated using dose and RSF.

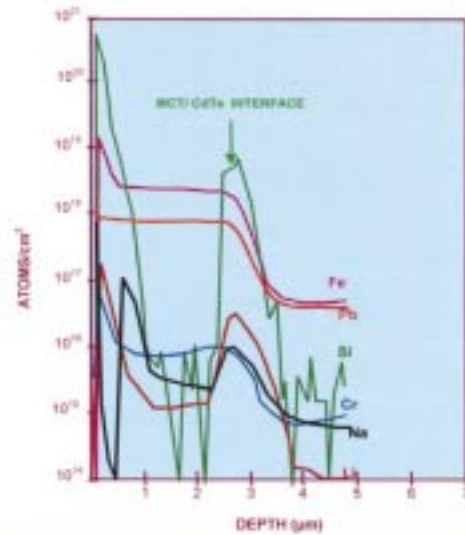


Figure 10. Interface concentration enhancement was observed in case of heterovalent impurities depth profiled across MCT/CZT.

Zinc Telluride substrates (CZT) is presented here. The main issues addressed are the identity, concentration and origin of various impurities. This work has been extensively discussed and published¹⁸. Depth profiling results were compared for epilayers/substrates prepared in-house, with imported epilayers/substrates using an oxygen ion beam, Fig. 10. *Li*, *Na*, *Si*, and *Fe* were found to be some of the impurities present. The level of these impurities in SSPL grown CZT substrates were found to be comparable with the substrate regions of an imported epilayer. However, the impurity concentration in the epilayers prepared from Hg-rich melts by High Pressure Liquid Phase Epitaxy (HPLPE) was found to be relatively higher. Considerable Interface Concentration Enhancement (ICE) of *Li*, *Na* and *Si* was observed in HPLPE layers. With the low temperature heat treatment for Hg-saturation, a complete removal of the ICE for *Li* and *Na* and reduction in the level of the ICE for *Si* was observed. This result is consistent with earlier literature reports¹⁹. The concentration of *Fe* in the HPLPE epilayers was found to be high (in 10^{17} - 10^{18} cm^{-3} range) in general.

No ICE was, however, observed for *Fe* and the low temperature heat treatment was found to have a little effect on the *Fe* depth profile. The *Fe* depth profiles showed a higher concentration (almost by an order of magnitude) in epitaxial region compared to the substrate region of HPLPE layers. An interesting observation during this work was that ICE was observed mainly in case of impurities heterovalent with respect to the matrix, depth profiled across MCT/CZT²⁰. *Cr* and *Pb* were also investigated for this purpose and they were found not to show any ICE (Table 3).

Low temperature annealing, for removal of impurities like *Na* and *Li* which are most likely being introduced during wafer preparation / handling, may therefore be beneficial from device point of view. However, after a low temperature anneal, these impurities accumulate in the near surface

Table 3. The observed effects of various impurities

Impurities	Possible source	ICE	Chemical nature wrt matrix
<i>Li</i>	Handling	Yes	Heterovalent
<i>Na</i>	Handling	Yes	Heterovalent
<i>Si</i>	Quartz	Yes	Heterovalent
<i>Cr</i>	Metallic part of growth chamber	No	Isovalent
<i>Fe</i>	Metallic part of growth chamber	No	Isovalent
<i>Pb</i>	Welded joints	No	Isovalent
<i>Zn</i>	From CZT substrate	No	Isovalent

region of 2-3 μm . This region should be discarded by an appropriate etch out process.

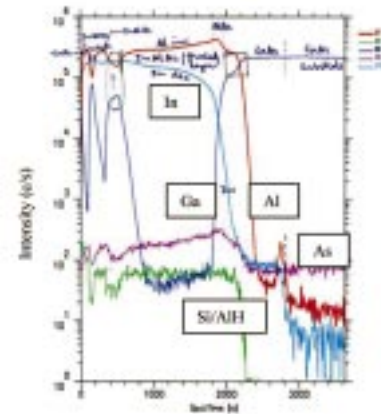
4.3 Identification and Estimation of Thickness of Various Layers in Multilayer Structures

SIMS is an extremely useful tool for identification, thickness determination and interface studies of the various layers of a multilayer structure. Quantum well lasers, quantum well photodiodes, MBE grown HEMT structures etc are some of the few important structures, having a diverse range of thickness, varying from a few nanometers to a few micrometers, which need to be depth-profiled for this purpose. Sputter-induced diffusion, Cascade mixing and Selective sputtering are some of the artifacts of sputtering which result in broadening of the depth profile of an originally sharp interface²¹. These effects are minimised by²²: (i) using low energy projectiles and (ii) increasing the angle of incidence. Best depth resolution is achieved by decreasing the primary ions impact energy to a minimum possible value and spatially filtering the secondary ions origin, to preserve only the ions that come from the flat crater centre region. Improved depth resolution would, however, mean reduced detection sensitivity.

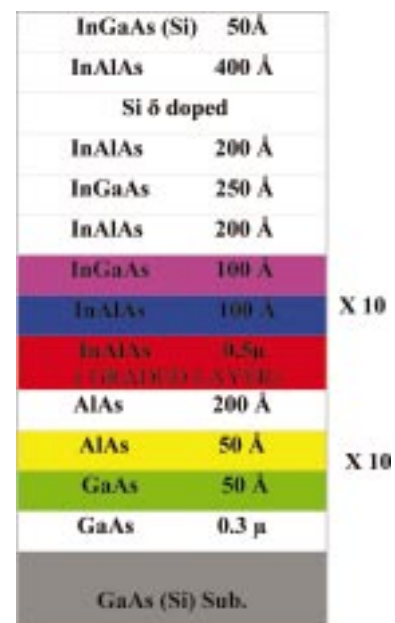
Figure 11(a) shows the depth profile of an MBE grown metamorphic structure, Fig. 11(b), identification of the various layers in the structure when it was analysed. Since a diverse range of thickness, varying from a few angstroms to the order of a micron need to be investigated, two sets of experiments were performed. Initially, an O^{2+} primary ion beam with an impact energy of 8.0 keV was rastered over an area of $250 \mu\text{m} \times 250 \mu\text{m}$ to provide a uniform primary ion current density. The secondary ions sputtering out of the sample surface were extracted with a voltage of 4.5 kV.

Only a fraction of the secondary ions, from a 150 μm diameter circular area centered in the rastered region, was accepted for analysis. This ensured the uniformity of sampling depth by using only the central flat bottom area of the sputtered out craters, thereby improving the depth resolution. The stability of the primary beam was about 1.5 per cent during the course of each depth profile.

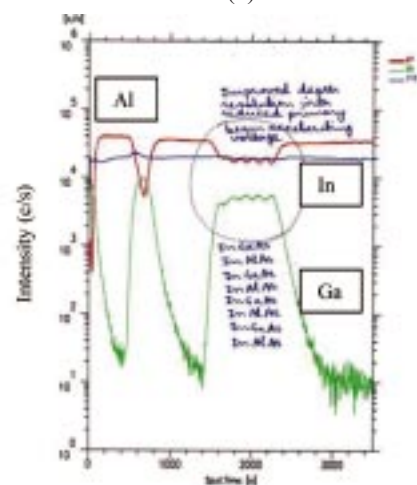
Though the thick layers are easily discernable, the encircled region, which comprises a super lattice structure, is seen only as a single layer. Fig. 11(c) shows a re-examination



(a)



(b)

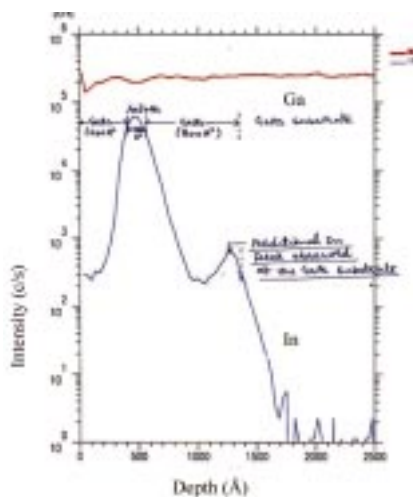


(c)

Figure 11. (a) Depth profile of a HEMT structure, (b) MBE grown metamorphic structure, and (c) Depth profile with reduced energy.

<i>GaAs</i> (402 Å)
<i>InGaAs</i> (150 Å)
<i>GaAs</i> Buffer (833 Å)
SI <i>GaAs</i> Substrate

(a)



(b)

Figure 12. (a) MOCVD grown single layer QW structure and (b) Depth profile of *In*.

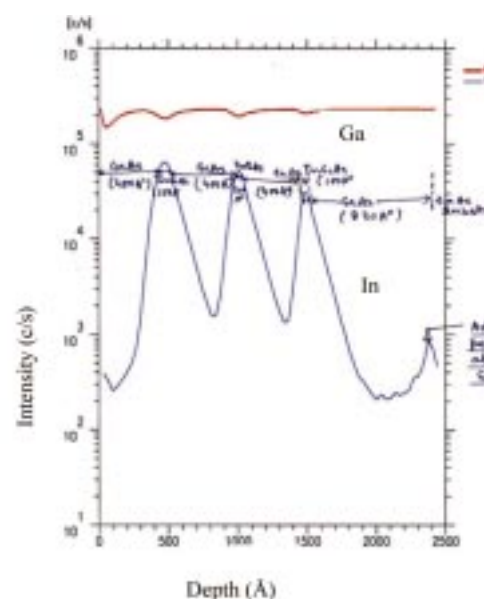
of this region, with reduced primary beam impact energy of 3.0 keV. Reducing the primary beam impact energy further improves the depth resolution. The encircled region of Fig. 11(c) clearly shows the expected 10 layers of alternate *InGaAs* and *InAlAs*, which were being seen as a single layer in Fig. 11(a).

4.4 Interface Examination

MOCVD grown *InGaAs* quantum well on *GaAs/n+GaAs* substrate structures were analyzed from the point of interface examination. Figure 12(b) is the depth profile of *In* in the structure of Fig. 12(a), grown by MOCVD. An unexpected small peak in addition to the expected 150Å layer of *InGaAs* was observed. Since the additional peak was observed to be away from the *InGaAs* layer, it was felt that *In* was appearing at the *GaAs/n+GaAs* interface. This was actually confirmed on converting the time axis to depth axis. The source of *In* was, however, yet to be ascertained. To investigate the source of this small peak, a three layer *InGaAs* QW on *GaAs* was analyzed Fig. 13(a) & (b) shows the *In* depth profile for this structure. Again a single additional peak slightly separated from the main peak was observed. The *In* peak was again found to be at the *GaAs/n+GaAs* interface. Depth measurements indicate that the additional peak is appearing only at the *GaAs/n+GaAs* interface and not appearing after every *InGaAs* layer. To establish the source of this additional peak, a structure grown using a clean susceptor was analyzed. Fig. 14 reveals that the small additional peak was no longer present. It can be safely concluded that the

<i>GaAs</i> (402 Å)
<i>InGaAs</i> (150 Å)
<i>GaAs</i> (402 Å)
<i>InGaAs</i> (100 Å)
<i>GaAs</i> (402 Å)
<i>InGaAs</i> (50 Å)
<i>GaAs</i> Buffer (833 Å)
SI <i>GaAs</i> Substrate

(a)



(b)

Figure 13. (a) MOCVD grown three layers QW structure and (b) Depth profile of *In*.

unwanted small *In* peak was due to some traces of *In* present in the susceptor from the prior growth.

4.5 Process Development / Failure Analysis

SIMS is an extremely useful technique for optimisation of growth parameters, effect of various process related heat-treatments²³, etc. during the process development stage. Typical examples of SIMS being used to investigate intermixing due to various process related heat treatments in thin multilayer metallic *Au/Pt/Ti* structures for Schottky contacts¹⁵ on *GaAs* crystals for MMIC applications is reported here. The thickness of individual layers in these applications is typically in the range of few hundred angstroms only. *Au/Pt/Ti* test structures, with 1000Å/300Å/300Å respective layer thickness were prepared on *GaAs* substrate by e-beam deposition process for the purpose of Schottky contacts for MMIC applications²⁴. Different samples were heat treated

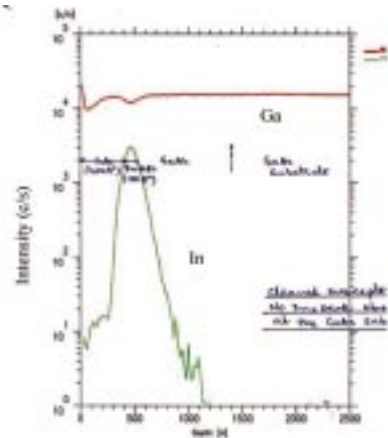


Figure 14. In profile with a cleaned susceptor.

at 400 °C and 500 °C by furnace annealing for 30 min. Figure 15(a) shows the depth profile of an as grown Au/Pt/Ti structure on GaAs. The stability of these contact structures against elevated temperatures encountered during device processing and their durability during device operation is a matter of concern. A comparison of Fig. 15 (a) and Fig. 15(b), where the sample has been annealed at 400°C, reveals that apart from a little diffusion of Au into the Pt layer and some As depletion from the near surface region

of GaAs, no significant intermixing of different components have been observed. On the other hand, a significant interlayer intermixing has been observed in case of 500 °C annealed sample, Fig. 15(c) and (d). On physical examination, too, the sample when heated up to 500 °C, appears partially blue whereas rest of the sample had the usual golden finish. The variation across the surface has been investigated by carrying out depth profiles in the two regions. The profile from the golden region, Fig. 15(c), shows some intermixing of the various layers. The Ti peak no longer exists and a substantial out-diffusion of Ti and As into the golden region is observed. The Pt peak is well pronounced but is pushed inside by ~ 150Å, towards the GaAs region. The profile from the blue region, Fig. 15(d), however, shows a complete mixing up of various elements. Profiling shows the barrier Pt layer to be completely diffused. The Pt peak no longer exists; hence allowing in-diffusion of Au into GaAs and out diffusion of As. The blue appearance further indicates the existence of a reacted layer. The study clearly shows that such structures are stable up to temperatures around 400 °C.

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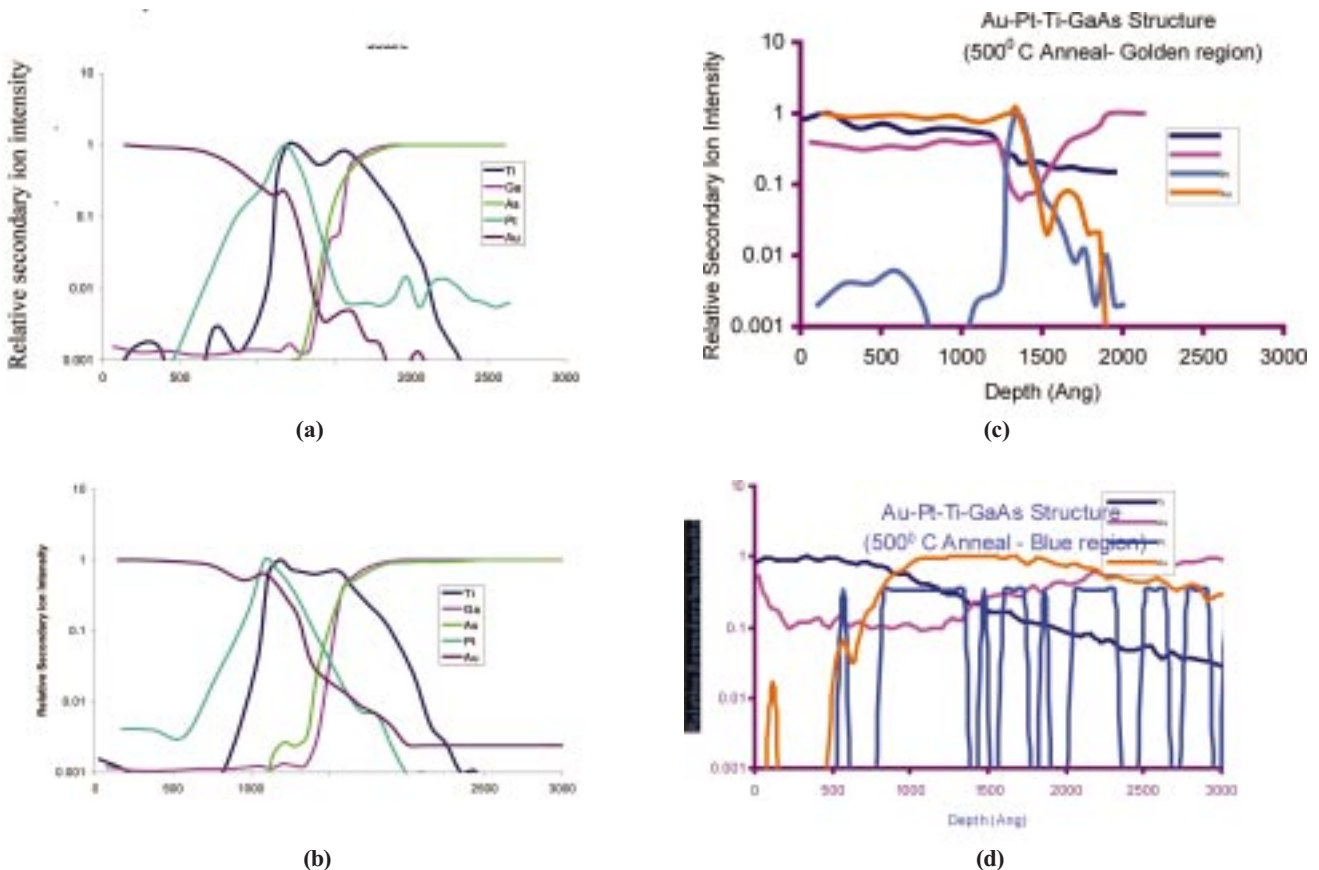


Figure 15. (a) Depth profile of a Au/Pt/Ti as grown structure on GaAs, (b) depth profile of a Au/Pt/Ti structure furnace annealed at 400 °C, (c) depth profile of a Au/Pt/Ti structure furnace annealed at 500 °C–golden region, and (d) depth profile of a Au/Pt/Ti structure furnace annealed at 500 °C–blue region.

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Contributors

Mrs Anuradha Dhaul obtained her MPhil (Physics) from Panjab University, Chandigarh in 1984. She joined Solid State Physics Laboratory (SSPL), Delhi in 1987. She is currently working on the characterization of materials and device structures using secondary ion mass spectrometer.

Mr S.K. Sharma obtained his diploma in mechanical engineering from Board of Technical Education, Delhi. He joined DRDO in 1989 and is currently working on SIMS characterization.

Dr R.K. Sharma obtained his MSc and PhD in physics from Banaras Hindu University. He Joined SSPL in 1982 and is currently heading the activity on infrared materials and devices. His areas of interest are: Preparation and characterization of semiconductor materials and device structures.

Dr Ashok Kumar Kapoor obtained his MSc from Agra University and PhD from Delhi University. Joined SSPL, Delhi in 1985 as Scientist 'B'. Working on characterization of materials using the electron microscopy and microanalysis and related techniques. Also worked on the study of conduction mechanism in conducting polymer materials. He has published about 20 papers in various journals and symposium. Presently Group Head of the characterization division