# A Novel Sample Structure for the Measurement of Indium Segregation Profiles in GaAs/InGaAs/GaAs Heterostructures

A. Loykaew, B. F. Usher, R. T. Jones, and P. J. Pigram

***Abstract*—A novel technique to measure In segregation profiles is proposed by which depth composition information is converted to surface composition information by creating a miscut surface which intersects a previously grown GaAs/InGaAs/GaAs heterostructure. The surface chemical profiles were measured by static Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS) and High Resolution X-ray Diffraction (HRXRD) measurements were made to allowdetermination of the thicknesses of all the layers in the structure and the profile of the miscut surface. The translation from vertical to horizontal coordinates could then be made with acceptable precision. In segregation was clearly observed and appears as an approximately exponential profile at both the GaAs/InGaAs and InGaAs/GaAs interfaces.**

***Index Terms*—In segregation, III-V compound semi-conductors, ToF-SIMS, HRXRD, MBE.**

## I. Introduction

Segregation is usually observed in compound semiconductors when a heteroepitaxial layer is grown in which one of the constituent atoms is either significantly larger or smaller than the substrate atom it replaces. As an example, when GaAs/InGaAs/GaAs heterostructures are grown by MBE, Inphysisorbs from the vapour and is subsequently chemisorbed [1], however surface segregation can return chemisorbed atoms to the physisorbed state, from which they can be chemisorbed again. This process leads to asymmetric and indistinct interfaces which can impact on device performance [2].

In surface segregation has previously been mostly studied by three methods. The first compares the average In composition in a particular layer, for example as measured by HRXRD, with the In composition in the surface as measured by a surface sensitive technique such as X-ray Photoelectron Spectroscopy (XPS), Auger Electron Spectroscopy (AES) or Ultraviolet Photoelectron Spectroscopy (UPS) [3]-[5]. This approach sometimes involves interrupting the growth process periodically to allow measurement of the chemical composition at the changing position of the surface. The second method requires creation of a cross-section specimen after which transmission electron microscopy (TEM) is used to create images of columns of atoms running parallel to heterointerfaces, from which information about the average composition within the columns can be extracted [6]. The third process also involves cleaving through a heterostructure to produce a cross-section sample, afterwhich the chemical composition profiles across interfaces are measured usinga surface sensitive technique, such as Energy Dispersive X-ray spectroscopy (EDX) orCross-sectional Scanning Tunnelling Microscopy (CSTM) [7]. The first method allows surface segregation to be detected but does not give detailed information about the shape of the segregation profile. It also suffers from the problem of finite sampling depth of the order of the characteristic length associated with the segregation process. The second method involves a complex analysis of TEM images, with the disadvantage that the test structures required to validate the approach cannot themselves be characterised satisfactorily. The third method suffers from the problem of finite spot size which is generally significantly greater than the characteristic segregation length. The purpose of the present study is to better understand the segregation process, using a new technique which doesn't require any growth interruptions and is not compromised by untested analytical complexity or by spot size or sampling depth effects,which can entirely mask the segregation process.

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The authors are with La Trobe University, Faculty of Science, Technology and Engineering, School of Engineering and Mathematical Sciences, Department of Electronic Engineering, Bundoora, Victoria 3086, Australia (e-mail: aloykaew@students.latrobe.edu.au, B.Usher@latrobe.edu.au, R.Jones@latrobe.edu.au, P.Pigram@latrobe.edu.au).

## II. Muraki's Theory of Segregation

There are many models of the segregation process, but most are based on the model developed by Muraki et al. in 1992 [8]. They measured the In segregation length in two types of MBE-grown $In_{0.126}Ga_{0.874}As$ QWs, one with different well thicknesses grown on GaAs substrates at constant temperatures of 643 K or 793 K and a second series of samples containing QWs with a constant well width of 4.6 nm grown at various substrate temperatures between 643 K and 893 K. Using data from Secondary Ion Mass Spectrometry (SIMS) and Photoluminescence (PL) measurements, an In segregation model was proposed as described below.

For a quantum well in which the first bilayer (one layer of group III and one layer of group V atoms in a III-V compound material grown on a (100) surface) is identified by $n$=1 and the last bilayer by $n$=$N$, the x-fraction of the segregating species in the $n^{th}$ layer is given by:

$$x_n = x_o(1 - R^n) \qquad 1 \le n \le N \tag{1}$$

for the Quantum well and

$$x_n = x_o(1 - R^n)R^{n-N} \qquad n > N \tag{2}$$

for the subsequent Barrier layer,
where $x_o$ is the in composition after the surface population of the segregating species achieves equilibrium, $R$ =exp(-$d$/λ) is the fraction of the top chemisorbed In atoms segregating back to the physisorbed state,d isthe thickness of one bilayer or half the lattice constant of GaAs (≅0.283 nm),λ is the segregation length or 1/e decay length,as determined from a measurement such as a SIMS depth profile, andN is the well width in bilayers.

Simulating Muraki's model for an $In_xGa_{1-x}As$ SQW, with $x_o$= 0.4, $R$ = 0.81 and $N$ = 35 bilayers, the composition profile obtained is shown in Fig. 1.

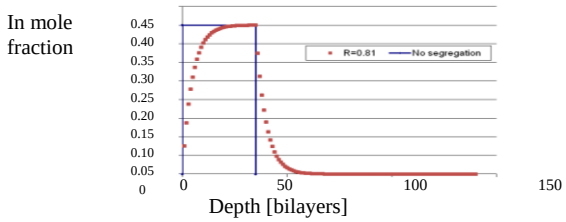



Fig. 1. Surface segregation following Muraki's model.

The main shortcoming of this model is that the segregation fraction R has no temperature or growth rate dependence, which is unlikely, however the probable reason for these shortcomingsis that there has been a dearth of experimental evidence to provide guidance to the development of theoretical segregation models. The approach taken here has been to develop a technique to create a surface miscutthat allows vertical chemical profiles to be translated into horizontal chemical profiles. The surface miscut is achieved by the growth of a GaAs wedge cap layer, which is subsequently uniformly polish etched to movethe surface so that it intersects the layers and interfaces of interest. This surface is then chemically profiled using ToF-SIMS and translated to a vertical profile using the HRXRD measurements of the profile of the miscut surface following growth

## III. Experimental Details of Sample Preparation

### A. Indium Profile Study Samples

Prior to the growth of each sample, GaAs (001) substrates were degreased and chemically polished with $H_2SO_4$:$H_2O_2$:$H_2O$ in the volume proportions 6:1:1 and transferred to the MBE growth chamber. Hydrocarbon contaminants and surface oxides were removed from the substrates by thermal cleaning to about 873 K. A typical structure is shown in Fig. 2. At its heart is a 300 nm InGaAs single layer grown epitaxially between a reference InGaAs/GaAs MQW and two sets of AlGaAs/GaAs marker layers. To complete the structure, a wedge-shaped GaAs cap layer was grown.

The growth rates used for the $In_{0.006}Ga_{0.994}As$ layer when the sample was in the normal growth position were 0.20, 0.45 and 1.00 μm/h and the substrate temperatures used were 713 K, 768 K and 833 K. Nine samples were generated, covering all the possible combinations of these growth rates and substrate temperatures.

All effusion cells were focused onto the substrate holder when in the normal growth position, resulting in uniform In, Ga and Al flux distributions across a10 mm × 10 mm area. All layers except the GaAs wedge cap at the surface were grown with the substrate holder in this position. To achieve the desired wedge shape in the final GaAs cap layer it was necessary to grow on substrates measuring 10 mm x 30 mm, however the In and Ga fluxes are not uniform along such a length. The purpose of the 10 period InGaAs/GaAs MQW was to allow determination of the In and Ga fluxes, and therefore the thicknesses of the InGaAs layer of primary interest, as a function of position along and across a sample. This flux distribution at larger distances from the centre of the substrate holder was determined by growing an InGaAs/GaAs MQW on a 10 mm x 50 mm substrate in the normal growth position and applying HRXRD analysis to find the In and Ga fluxes on a 5 mm mesh grid across the surface.

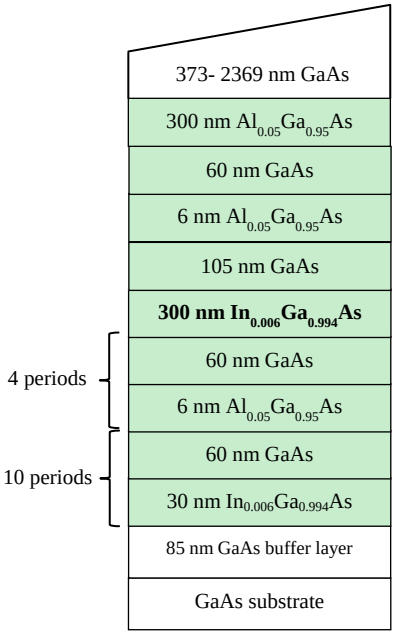



Fig. 2. Sample structure containing reference InGaAs/GaAs MQW, AlGaAs/GaAs marker layers, the InGaAs layer of interest, a 300 nmAlGaAsvisualisation layer and the GaAs wedge shaped cap layer.

The purpose of the AlGaAs/GaAs MQW was to again use HRXRD to determine the Al and Ga flux rates so that the thicknesses of all the AlGaAs and GaAs marker layers would be known as a function of position across the sample surface.

Since measurement had shown that the minimum flux at the substrate holder edge was only about 70 per cent of the flux at the centre when in the normal growth position, another strategy had to be developed to increase the ratio of the thicknesses at the centre and the edge of the substrate holder for the purpose of creating a suitable GaAs wedge on the surface. For the growth of this layer, the substrate holder was rotated by 23° around a vertical axis from the normal growth position to achieve a minimum flux at the furthest end of a 10 mm × 50 mm sample of only 5% of that at the end nearest the normal growth position. The normal and 23° rotated geometries are shown in Fig. 3.

The cap-layer shape had to be controlled to achieve a slope of less than $2.825 \times 10^{-4}$, so that one pixel in the ToF-SIMS image would be no larger than the width of a

single atomic terrace between surface steps, but steep enough to cut through all the interesting layers and interfaces. Flux distributions were studied by growing 30-period $In_{0.05}Ga_{0.95}As$/GaAs multi quantum wells on 10 mm wide and 50 mm long GaAs substrates set at an angle of 23° from the normal growth position with the long axis of the sample aligned horizontally. HRXRD was performed using a PanalyticalX'pert PRO materials research diffractometer equipped with a Cu point focus source, parabolic multilayer mirror and a Bartels style four-bounce Ge(220) monochromator. Rocking curves were collected at four azimuthal PHI angles of 0°, 90°, 180° and 270° to detect layer tilting and allow for its correction in the analysis. A self-consistent method of analyzing MQW structures, which uses MBE shutter timing information, allowed all layer thicknesses and compositions to be determined[9]. HRXRD measurementsover a 4.5 mm × 5.0 mm grid allowed determination of individual group III growth rates at positions across and along the sample.

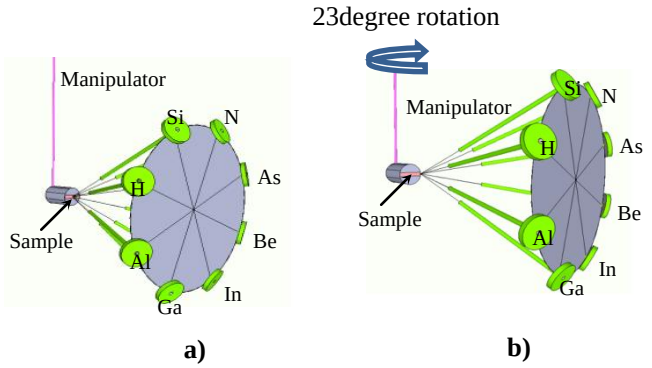



Fig. 3. a) MBE growth with the sample in the normal position and b) growth with the sample rotated 23 °clockwise around a vertical axis.

Examples of the HRXRD spectra obtained are shown in Fig. 4 where changes in the MQW satellite peak periodicity at positions 10 mm apart allow MQW layer thicknesses and compositions to be determined both across and along the samples.

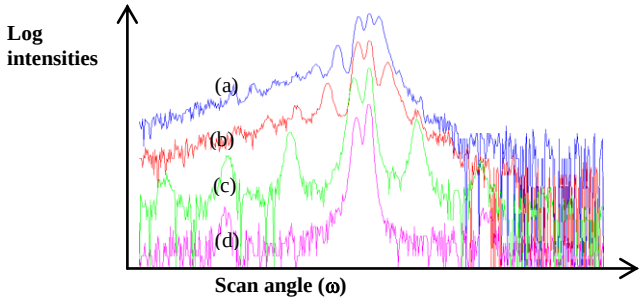



Fig. 4. Experimental HRXRD spectra collected along the center line at points 11mm, 21mm, 31mm and 41 mm from the thick end for (a), (b), (c) and (d) respectively. The spectra have been shifted vertically for clarity.

Fig. 5 a) shows the wedge cap layer thicknesses every 5 mm on three parallel lines along the sample length positioned at the center and 4.5 mm apart. Fig. 5 b) shows the Ga fluxes determined from analysis of an InGaAs/GaAs MQW structure at the same positions at which cap layer thicknesses were determined. It can be seen that the left profile had the lowest flux when compared with the others. This two dimensional flux variation meant that the wedge cap had position-dependent slopes both along and across the samples.After the flux distribution had been determined, the wedge cap layer gradient could be manipulated by changing the growth time. The cap profile shown in Fig. 2 is representative of what could be achieved, with a thickness ratio from the thick to the thin end of about 6.4.

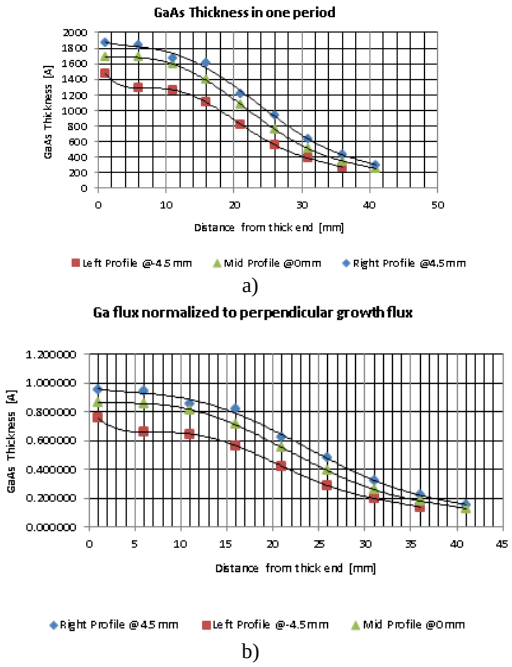



Fig. 5. Profile of a) GaAs wedge cap layer thicknesses and b) Ga fluxes obtained from an analysis of an InGaAs/GaAs MQW structure.

Samples were then chemically etched with a dilute polishing etch ($H_2SO_4$:$H_2O_2$:$H_2O$ in volume proportions 1:1:70) to a depth of approximately 147 nm such that the AlGaAs layers became visible and the InGaAs layer was estimated to be positioned at the sample center. Approximately four liters of this etchant was prepared and a "flow-etch" approach taken, whereby etch products were more likely to be removed rather than deposit onto the surface. Earlier experiments in which this procedure was not followed resulted in significant etch product contamination across the surface which compromised later ToF-SIMS measurements. This process was aided by the fact that exposed AlGaAs bands can be viewed by eye which allowed the etch process to be stopped when the InGaAs band was at the center of the sample. Subsequently, samples were soaked in $H_2SO_4$:$H_2O$ in volume proportions 1:1 followed by a HCl rinse for two minutes to remove hydrocarbons, residual etch products and oxide layers before transferring them to the ToF-SIMS chamber. Secondary-ion mass spectra and elemental maps were acquired with a ToF-SIMS IV instrument (ION-TOF GmbH, Münster, Germany) equipped with a bismuth liquid-metal ion gun as the primary-ion source and a reflectron time-of-flight analyser. For each acquisition, a primary-ion beam of 25 keV Bi+ ions was pulsed over a 100 µm x 100 µm field of view in high-current bunched mode below the static limit. Larger images from 500 µm x 500 µm up to 8 mm x 18 mm were obtained as tesselations of these fields of view by rastering the stage with a 1 pA primary-ion current and positive ion polarity. The pressure in the instrument's analysis chamber was less than 1 x $10^{-9}$ mbar.

Having developed a strategy for creating a wedge shaped

sample where the surface intersects all the layers and interfaces of interest, a number of measurements were made to confirm the sample specifications were satisfactory and to characterize the depth resolution of the ToF-SIMS technique. These included confirming that the miscut surface intersected the layers of interest, that the surface roughness following etching was similar to that before etching and that the ToF-SIMs sampling depth was known. The additional experiments performed to clarify these important aspects of the sample preparation approach taken are described below.

### B. *Sampling Depth Sample*

A δ–doped Si sub-monolayer was deposited on a GaAs surfaceat a low substrate temperature of 723K to avoid segregation or diffusion effects. Substrate holder rotation around the surface azimuth provided a reasonably uniform incident Si flux during this deposition which was followed by the growth of two bilayers of GaAs to lock in the Si. A wedge shaped GaAs cap layer was then grown by moving away from the normal growth position by 23° as explained earlier. This resulted in a wedge that was two bilayers thick at the thin end and 40 bilayers thick at the thick end.

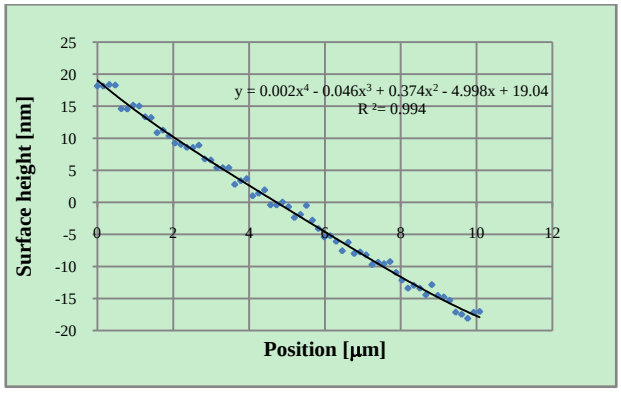
### C. *Etched Profile Sample*

A 15 period 0.3 nm $In_{0.40}Ga_{0.60}As$ and 0.9 nm GaAs MQW was grown on a 10 mm × 30 mm GaAs substrate at a substrate temperature of 813 K and was capped by a wedge shaped cap layer. This sample was chemically etched and cleaned beforebeing transferred to the ToF-SIMS chamber for analysis.

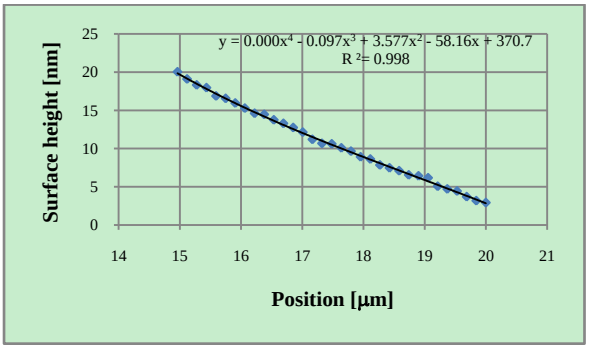
## IV. Sample Characterization, ToF-Sims Sampling Depth and Segregation Measurements

### A. *Surface Roughness Measurements*

Roughness measurements of the etched surfacewere made using Atomic Force Microscopy (AFM) over a20 µm × 20 µm area to check whether the dilute polishing etch had increased the surface roughness.Fig. 6 shows one scan line of AFM data from etched and non-etched surfaces collected with 128 × 128 scan point resolution. The curves shown were best fit 4th order polynomials and the root mean square calculations, which were 0.79 nm and 0.18 nm respectively, show that there was an increase in the surface roughness between the etched and non-etched surfaces but the horizontal scale of increased roughness is small compared with the ToF-SIMS pixel size.




a)

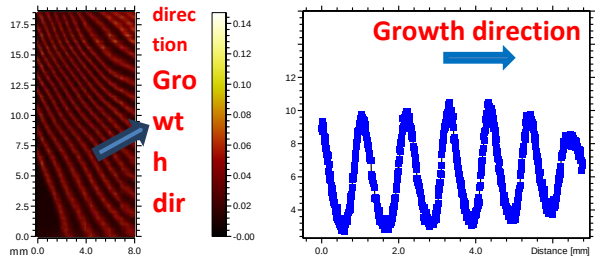



b)

Fig. 6. The surface height of a) etched and b) non-etched surfaces fitted by polynomials of 4th order.

### B. *ToF-SIMS Measurements to Profile the Etched Wedge*

The Static ToF-SIMS used a Bi+ primary ion total dose of less than $10^{13} ion \cdot cm^{-2}$with typical pulse lengths less than a few nanoseconds [10]. To ensure that the etched surface followed the wedge cap profile, ToF-SIMS images of etched samples showing continuousIn bands could be compared with the known wedge profile.TheToF-SIMS surface image inFig. 7 a) shows anIn+ peak intensity image across an8 mm × 18 mmetched surface where the etched wedge profile cut through the MQWstructure. It can be seen that the In bands are diagonal across the surface and each In band is non-linear as a consequence of the two dimensional flux variation present during growth of the wedge cap.




a) b)

Fig. 7 a) Bands of In+peak intensities normalized to total ion intensities imaged by ToF-SIMS over a 8 mm × 18 mm scanned area. The imageis a composite generated from 16 × 36 = 576 patches of size 500 µm × 500 µm with 100 pixels per mm resolution. b) In profile along the sample length.

Subsequently the structures shown in Fig. 2 were prepared and the In and Al profiles were measured across the $In_{0.006}Ga_{0.994}As$ band including the GaAs layers on either side of this band and the AlGaAs marker layers. From these scans all layers could be identified and from their known thicknesses the surface profile could be plotted as a function of horizontal and vertical position across the entire sample surface.

### C. *Sampling Depth Investigation*

Fig. 8 shows aschematic of a δ–doped Si bilayer embedded in a GaAs layer. Static ToF-SIMS sampling depth measurements were performed by acquiring spectra on 100 µm × 100 µm areas with 128 × 128 scan point resolution along the wedge cap layer from the thin end to the thick end. Silicon intensities were expected to decrease to background levels when the GaAs cap thickness was equal to or greater than the sampling depth.

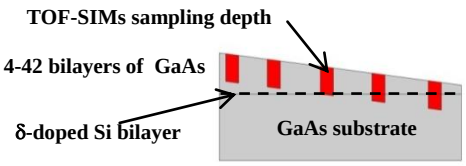



Fig. 8. Sampling depth sample with wedge shaped GaAs cap layer on a δ–doped Si layer grown at a substrate temperature of 723K.

Fig. 9 shows the $Si^+$ peak intensitydecreases to background levels at a wedge cap thickness of about 4.4 bilayers, which was concluded to be the maximum ToF-SIMS sampling depth. This is much less than the measured segregation lengths, confirming that sampling-depth effects donot dominate the observed segregation profiles.

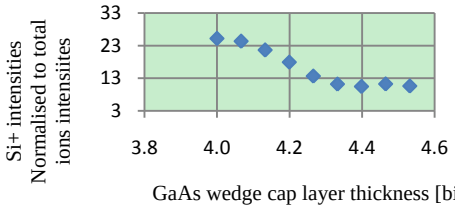
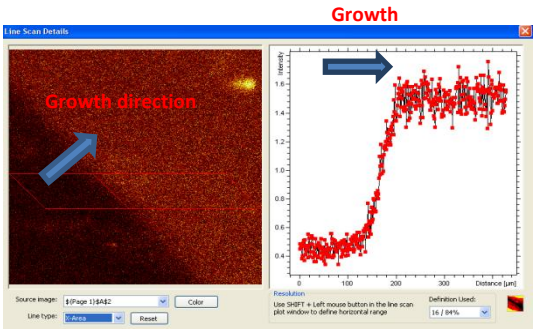


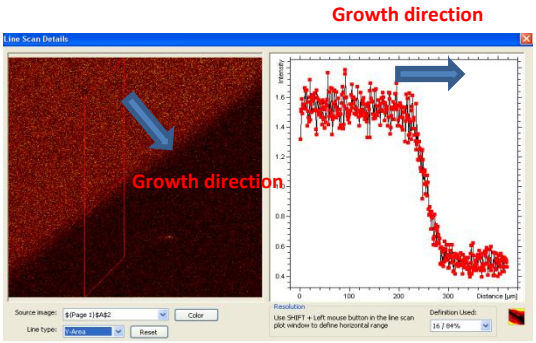

Fig. 9. Si intensities obtained from a wedge shaped GaAs cap layer grown on top of a δ-doped sub-monolayer Si layer as a function of position along the sample.

### *D. Surface Segregation Measurements*

Figs. 10 a) and b) show $In^+$ intensity profiles at the rise and decay interfaces of a 300nm $In_{0.006}Ga_{0.994}As$ layer situated between GaAs layers and grown at 713 K. The group III growth rate for the InGaAs layer was 0.45 µm/h and a 500 µm × 500 µm area was scanned with 1000 pixels per mm resolution. To optimize beam focusing, the scanned area size was minimized to eliminate peak shift effects due to surface-height variation which can cause misleading results.



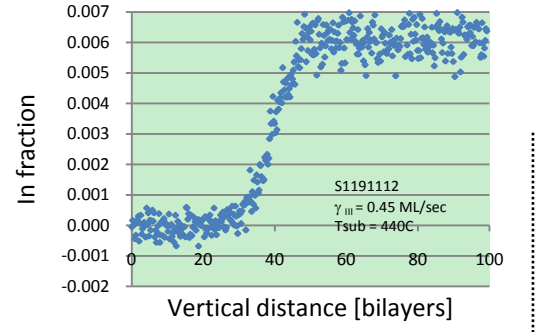

a)



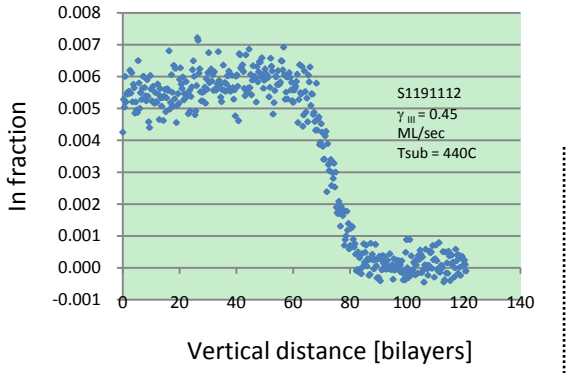

b)

Fig. 10 a) Image of $In^+$yield normalized to total ion yield and b) image of vertical mirrored $In^+$ yield normalized to total ion yield with the $In^+$yield profile shown as a function of horizontal distance at the rise and decay interfaces.

Figs. 11 a) and b) are In fraction profiles as a function of vertical depth across the rise and decay regions. $In^+$ yields have been converted into mole fractions and the translation of horizontal to vertical position was possible because the surface profile was known. The In fraction rose to reach an equilibrium value and then decayed after the In shutter was closed.




a)




b)

Fig. 11. a) and b) are in fractions in the rise and decay regions as a function of vertical distance.

The rise and decayInmeasurement data was averaged every five data points to give one bilayer depth resolution. The transition region of the profiles were fitted by 3th order polynomials to generate a smoothed representation of the measurement data. The turning points of these polynomials were matched to plateaus at the background and equilibrium in levels to complete the profile.

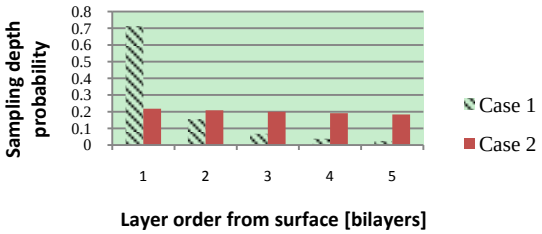
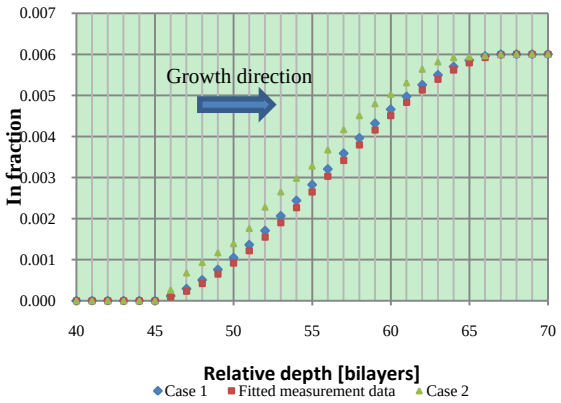



Fig. 12. Sampling depth probability from the top five bilayers. Case 1 assumes that more than 90% of the signal originates from the first two bilayers whereas case 2 shows a slight decrease in acquired signal probabilities from the top most bilayer to the fifth bilayer.

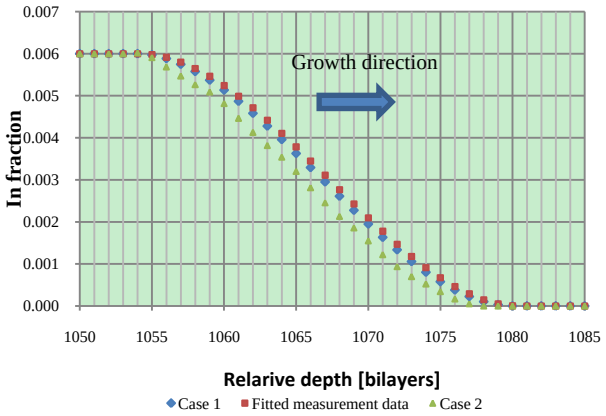
In order to eliminate sampling depth effects from the smoothed measurement data, a process of deconvolution over a5 bilayers sampling depth was performed. We used 5 bilayers instead of the measured 4.4 bilayers obtained from the sampling depth experiments to give a margin of safety to this procedure.Two extreme cases were considered for the

sampling probabilities, case 1 for which the signal is predominantly from the top two bilayers and case 2 for whichthe signal originates almost equally from each of the five bilayersas shown in Fig. 12.

The in profilesextracted after taking sampling depth effects into account are plotted in Fig. 13. It is clear that there is little difference in the In profilesobtained for the two extreme sampling depth probability cases tested.




a)




b)

Fig. 13. a) and b) show theIn rise and decay measurement data fitted by 3rdorder polynomialsand two solutions for the segregation profiles extracted after taking into effect sampling depth effects.

These measurements clearly meet the objectiveof defining the segregation length and In profiles at SQW interfaces down to the bilayer scale. The dependence of these parameters on the group III growth rate and substrate temperature are the subject of ongoing work.

## V. Discussion and Conclusions

A novel approach to convert vertical composition profiles into horizontal profiles by the creation of a well characterised shallow wedge has been presented. The horizontal compositional profile of a segregating species has been measured by static ToF-SIMS and the effect of averaging over a depth into the surface has been quantified by determining the sampling depth relevant to our growth conditions. The flow etchant approach to creating a wedge through the layers of interest was an important technique which preventedetch products depositing backonto the surface.

The observed in segregation profiles measured by ToF-SIMS are in general agreement with Muraki's model. Future work to test the role of substrate growth temperature and bilayer growth rate in the segregation process should lead to a more detailed description of the segregation process.

## Acknowledgment

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**Aphirak Loykaew** received an undergraduate Civil Engineering degree from KhonKaenUniversity in 1998 and his first class honours Master degrees in Nuclear Engineering from Chulalongkorn University, Thailand in 2005. He became a lecturer in the Physics Department, UdonthaniRajabhat University,Thailand and has been teaching lecture courses in Nuclear Physics, Physics I, Physics II and conducting modern Physics laboratories. His research area is naturally occurring radioactive materials (NORMs) in the petrochemical industry and he is also interested in material science. He was granted a PhD scholarship to attend La Trobe University to study semiconductor materials.

**Brian Usher** was born and educated in Perth, Western Australia, graduating with aB.Sc (Hons) (1971), Dip.Ed (1972) and PhD (1981) in physics from the University of Western Australia. He has worked in an industrial research lab and several universities in Australia and spent three years working in the UK and South Africa on materials science research projects. He has published in the fields of the nucleation and growth of thin films, semiconductor growth byMBE, x-ray diffraction and topography, transmission electron microscopy and dislocation dynamics.

**Robert Jones** completed his B.Sc. (Hons) degree at the University of Adelaide, Adelaide, South Australia, in 1986. After working as a chemistand then a surface analyst, he enrolled as a PhD candidate at the University of South Australia, Adelaide, Australia, studying theelectronic structures of sulfide minerals. Since the award of hisPhD in 2007, he has been working as a Post–doctoral Research Fellow in the Centre for Materials and Surface Science at La Trobe University, Melbourne, Australia. His current field of research is the chemical characterisation of material surfaces by X-ray photoelectron spectroscopy and static secondary-ion mass spectrometry.

**Paul Pigram** completed a B.Sc (Hons) degree in1987, and a PhD in applied physics in 1991 at the University of Sydney. After a postdoctoral appointment at the University of New South Wales in the field of surface science, he took up a faculty position in physics at La Trobe University in 1995. Currently Paul is a Reader and Associate Professor in Physics at La Trobe University as well as Head of the Department of Physics. Paul is the Director of the Centre for Materials and Surface Science (CMSS), a centre hosting a world class surface analytical capability including XPS, ToF-SIMS, UPS, AFM (ambient), UHV SPM, contact angle analysis and a variety of physical characterisation resources. His research interests are directed to understanding interactions at surfaces, the creation of functional molecular structures including polymeric sensors, tailored surface chemistries and nanoparticles for drug delivery, and molecular characterisation of these surfaces.