

# The effect of the In concentration on the surface morphology of InGaAs-GaAs heterostructures grown by MBE on GaAs substrate

E Gómez-Barojas<sup>1</sup>, R Silva-González<sup>2</sup>, R M Serrano-Rojas<sup>1</sup>, M A Vidal-Borbolla<sup>3</sup>, M A Rodríguez-Moreno<sup>1</sup> and G Santamaría-Juárez<sup>1</sup>.

<sup>1</sup>CIDS-IC, Benemérita Universidad Autónoma de Puebla. Apdo. Postal 196, Puebla, Pue. 72000. México

<sup>2</sup>Instituto de Física, Benemérita Universidad Autónoma de Puebla. Apdo. Postal J-48, Puebla, Pue. 72570 México

<sup>3</sup>Instituto de Investigación en Comunicación Óptica. Universidad Autónoma de San Luis Potosí, Av. Karakorum 1470, Col. Lomas Altas 4<sup>a</sup>. Sección, San Luis Potosí, S. L. P., 78100, México

E-mail: egomez@ifuap.buap.mx

**Abstract.** A set of 3 heterostructures were formed by 10 periods of InGaAs-GaAs epitaxially grown on GaAs substrate by means of a molecular beam epitaxial system. Scanning electron microscopy (SEM) cross section images at high magnification show that the heterostructures present good periodicity. SEM micrographs of the surface morphology chemically etched show the coalescence effect of In due to an unequal etching rate of In and GaAs. Auger electron spectroscopy (AES) depth profiles show that the first GaAs layers in the 3 samples are off-stoichiometric and that the alloy layers present In square and triangular depth profiles.

## 1. Introduction

Since the decade of the 80s, considerable interest had been devoted to the growth of heterostructures based on lattice mismatch epitaxial layers in order to improve device performance and obtain new properties. In the case of strained  $(\text{InAs})_x(\text{GaAs})_y$  superlattices abrupt heterointerfaces are required because compositional fluctuations at heterointerfaces results in uncertainty in both composition and lattice constant [1]. These fluctuations are attributable to surface steps in each layer. The surface steps are composed of excess atoms, which remain on each layer because it is difficult to control the molecular beam intensities and the growth time precisely enough to form just one monolayer.

The aim of this work is to study exsitu the surface morphology, the periodicity and elemental composition of a set of 3 InGaAs-GaAs heterostructures grown on GaAs (100) substrate by a MBE system. The heterostructures are formed by 10 periods of InGaAs-GaAs epitaxially grown on GaAs substrate. The techniques used for this purpose are SEM and AES.



## 2. Experimental procedure

### 2.1 Preparation of $\text{In}_x\text{Ga}_{1-x}\text{As}$ -GaAs heterostructures

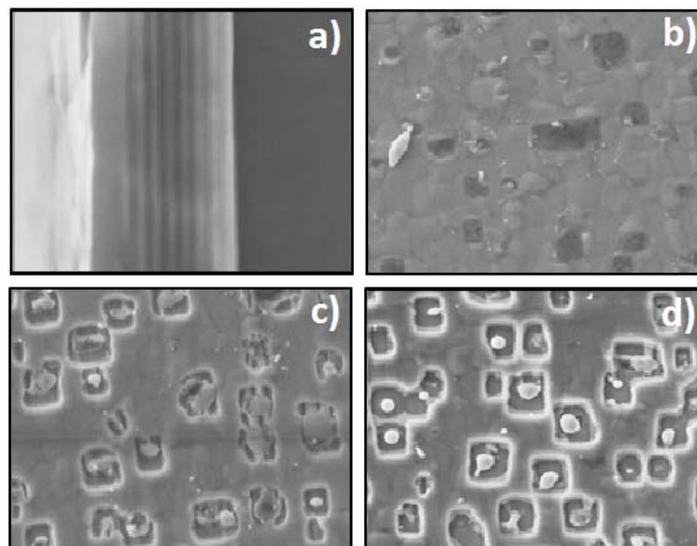
Deposition was carried out in a RIBER, MBE32 system equipped with a high energy electron diffraction (RHEED) facility. The GaAs substrates were semi-insulating with (100) orientation. The GaAs substrates were degreased chemically. To remove native oxide from the surface substrate, after loaded in the growth chamber a heat cleaning was performed at 580 °C and the RHEED intensity oscillations of C and O were monitored until obtaining only the GaAs pattern. The substrate temperature  $T_s$  was measured by an infrared pyrometer which was calibrated by using the Gallium oxide desorption temperature 580 °C as a reference.

The heterostructures are formed by 10 periods of  $\text{In}_x\text{Ga}_{1-x}\text{As}$ -GaAs epitaxially grown on GaAs substrates with nominal thickness of 500 and 1000 Å, respectively. Three different growth runs labeled A, B, C were done. The growth parameter for both GaAs and  $\text{In}_x\text{Ga}_{1-x}\text{As}$  layers of the three runs were: the molecular beam pressure (MBP) were: for  $\text{As}_4$ ,  $8.1 \times 10^{-6}$  Torr at 205 °C; for Ga,  $3.6 \times 10^{-7}$  Torr at 914.5 °C and for the alloy layers the MBP were: for In of growth run A,  $4 \times 10^{-7}$  Torr and 807 °C, for run B,  $2.1 \times 10^{-7}$  and 768 °C and for the growth run C,  $9.6 \times 10^{-8}$  and 731 °C. SEM observations were performed in a JEOL 5400LV system and composition analysis in a JEOL (JAMP 7800) AES equipped with a secondary electron detector and an Ar ion sputtering system.

## 3. Results and discussion

### 3.1 Surface morphology by SEM

Figure 1 (a) shows a SEM cross section view at X50000 of sample A. The grooves pattern was revealed by dipping the sample into a solution  $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$  (1:5:25) for 8 s. The etching rate is 400 Å/s for GaAs. Lighter fringes correspond to InGaAs, and the darker ones to GaAs layers. They show good periodicity, we could say good interface abruptness and the average thickness of the InGaAs and the GaAs layers determined from these SEM images, are in reasonable agreement with those obtained from the growth rate.



**Figure 1.** SEM micrographs of sample A: a) of a cross section, and of the surface morphology at 50000X: b) before etching, c) after 5 sec etching and d) after 8 sec etching.

Also figure 1 shows SEM micrographs of the surface morphology of the uppermost layer of sample A b) before etching; this morphology looks like it contains bubbles some of them already exploded (darker areas); c) after 5 s etching; this morphology presents square dislocation pits with a spherical particle in the pits crater [2] and d) after 8 s etching; at this time the morphology has been better revealed.

The as “grown” surface morphology of sample B does not present any particular texture. However, after 10 s of chemical etching, the surface morphology looks like some parts of the film were not completely removed. Also, the surface morphology of the “as grown” heterostructure C does not present any texture but after 5 s of chemical etching very small etch pits are developed with no particle in the pits crater, SEM images not shown here.

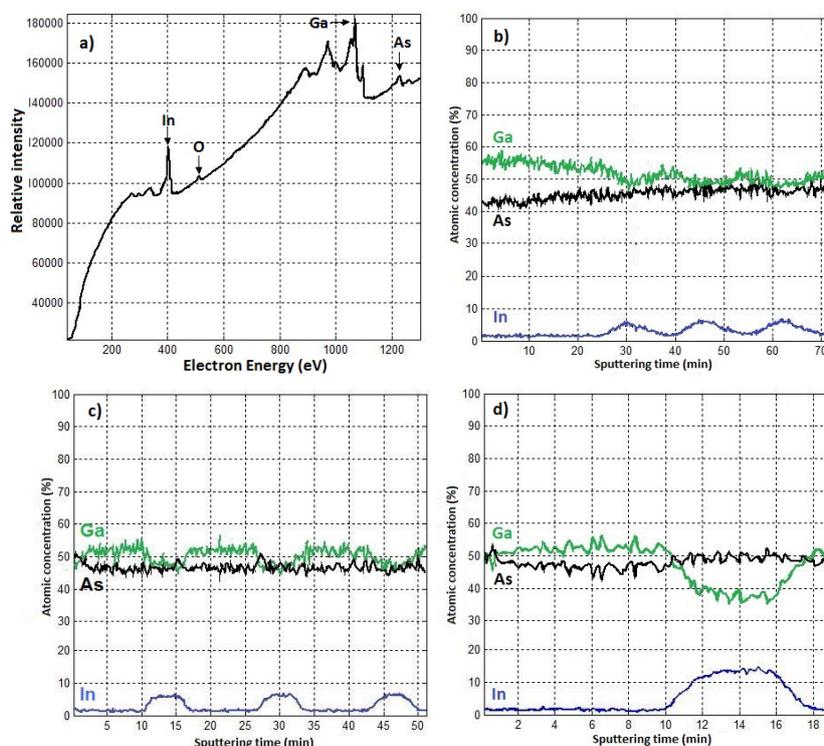
### 3.2 AES analysis

Direct Auger spectra in the spot mode were recorded from the surface morphology of sample A. figure 2a shows the spectrum recorded from a particle in a crater. The average relative concentration was calculated using the formula:  $C_i = (I_i/R_i)/\Sigma(I_j/R_j)$ , where C is the concentration, I is the measured intensity and R is a sensitivity factor, the subscript i represents the element in the sample [3]. Thus, a particle in the pits crater contains 21.4 at % In, 23.1 Ga, 48.6 at % of As and 6.9 at % of O. In a region between pits the composition is 4.1 at % In, 46.7 Ga, 43.1 at % As, and 6.2 at % of O. We infer that the In coalesces because the etchant employed is not effective for In. Furthermore, we checked the stoichiometry of a brand new GaAs wafer by AES. This sample underwent the same chemical etching process for 8 s. and immediately analyzed by AES. The surface composition was 10.3 at % O, 36.6 at % Ga and 53.1 at % As which means that during the etching process the H<sub>2</sub>O<sub>2</sub> used as an oxidizing agent causes Gallium oxidation mainly [4].

A personal computer program was done to calculate and plot the atomic percentage of the constituent elements of the 3 heterostructures from the Auger electron intensity signal vs Ar ion sputtering time spectra recorded by the Auger system [5]. These spectra are shown in figure 2 b, c, d, and were recorded from the “as grown” A, B and C samples. The Ar ion beam sputtering was operated at 3 keV and 25 mA of current. These depth profiles were recorded with similar measurement parameters for their later comparison.

The corresponding relative atomic percentage of elements was used to calculate the elemental mole fractions. The composition of the first layer of sample A is Ga<sub>0.66</sub>As<sub>0.34</sub> then, the following layers almost consist of a series of alloy layers with a triangular variation in In since the GaAs layers between the alloy layers are very thin. Thus, the composition at the ridge of the first triangle is In<sub>0.06</sub>Ga<sub>0.59</sub>As<sub>0.35</sub>. The Auger depth profile of sample B shows a square periodicity in the barrier-well structure. The composition of the first layer is Ga<sub>0.63</sub>As<sub>0.37</sub> and that of the second layer is In<sub>0.12</sub>Ga<sub>0.52</sub>As<sub>0.36</sub>. Finally, in the depth profile of sample C, the composition of the first layer is Ga<sub>0.63</sub>As<sub>0.37</sub> and that of the second layer is In<sub>0.25</sub>Ga<sub>0.40</sub>As<sub>0.35</sub>. From the Auger depth profiles data we infer that the first GaAs layers in the 3 samples are off-stoichiometric with similar values of mole fractions. The thickness of the first layer in B and C samples are equal since both were Ar ion etched in 10 min, nominally 1000 Å, while the first layer of sample A was etched in 25 min which would correspond to 2500 Å thickness. Analyzing the mole fractions of the first InGaAs layers in the three samples, we observe that the As mole fraction are very similar, this means that the under stoichiometric concentration for As is maintained in these alloy layers. Besides, since the In element is isoelectronic to Ga then the In atoms substitute the Ga atoms to form the alloy InGaAs. Now, the In mole fraction is the largest in C, smaller in B, and the smallest is the maximum mole fraction of sample A which as mention before show a triangular variation in concentration. Now, going back to the surface morphology of the chemically etched sample A (figure 1) we can make the following reasoning. Considering 400 Å/min as the chemical etching rate and 8 s of etching time, a GaAs layer of 3200 Å was removed chemically the corresponding surface morphology is shown in figure 1d. Now, according to the Auger depth profile of sample A the thickness of the first GaAs layer is about 2500 Å.

A layer of this thickness would have been removed chemically in 6.2 s, in the next 1.8 s the etchant certainly found a series of thin InGaAs alloy layers with a triangular variation of In concentration. Since the In element is more resistant than the GaAs to this etchant then it coalesced forming the small spherical particles observed in figure 1d.



**Figure 2** (a) Direct Auger spectrum of a particle in a crater of sample A. Auger depth profiles of the as grown  $\text{In}_x\text{Ga}_{1-x}\text{As}$ -GaAs heterostructures grown on GaAs: (b) of sample A, (c) of B and (d) of sample C.

In summary, from SEM cross section micrographs at high magnification it has been possible to observe the constituting layers of the  $\text{In}_x\text{Ga}_{1-x}\text{As}$ -GaAs multistructure grown on GaAs by an MBE system. Surface morphology SEM micrographs have shown the coalescence effect of In due to an unequal chemical etching rate of the etchant with respect to the one of GaAs. From Auger depth profiles we have inferred that the GaAs layers of the  $\text{In}_x\text{Ga}_{1-x}\text{As}$ -GaAs heterostructure deposited on GaAs substrate are off stoichiometric.

#### 4. Acknowledgments

Work supported by Benemérita Universidad de Puebla by means of a VIEP-Project and by Secretaría de Educación Pública: CA190, México.

#### References

- [1] Y Matsui, H Hayashi, and K Yoshida 1986 *Appl. Phys.* **1** 219
- [2] I Tarján and M Mátrai 1972 *Quality Control by Chemical Etching* p 74
- [3] *Handbook of Auger Electron Spectroscopy* (JEOL) 1982
- [4] D B J Lee 1969 *Appl. Phys.* **40** 4569
- [5] D Briggs and M P Seah 1990 *Practical Surface Analysis. Vol. 1 Auger and X-ray photoelectron spectroscopy.* (John Wiley and Sons)