Journal of Physics: Conference Series **480** (2014) 012017 doi:10.1088/1742-6596/480/1/012017

Microstructure AFM study and raman spectra of In-GaAs bilayers prepared by R.F. magnetron sputtering on Si(100) substrates

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Received 15 September 2013, in final form 18 Novembrer 2013

Abstract. Preliminary results of In-GaAs bilayers prepared by RF Magnetron Sputtering on Si(100) substrates are presented. The growth temperatures were 300 and 580 °C for the high purity targets of In and GaAs, respectively. Under an Ar atmosphere, three samples were prepared, each one with a GaAs buffer layer deposited for 60 minutes, then two cycles were prepared as follows: In layers from 5, 10, and 15 minutes with a subsequent GaAs layer deposited for 20 minutes. The morphological and optical studies of the samples were made by means of Amplitude Modulation Atomic Force Microscopy (AM-AFM) and Micro Raman Spectroscopy (μRAMAN). In order to analyze and correlate the surface morphology and alloy composition, the samples were cleaved along the [001] direction and subsequently characterized by AM-AFM and μRAMAN. From the topographic images, a statistical study of the roughness and grain size is made. Additionally cross sectional images were performed for each sample, from where the phase channel, which is sensitive to the material properties was of particular interest. The Raman spectra showed the two vibrational phonon modes related to the InAs and GaAs.

[1] Introduction

The Indium Gallium Arsenide (InGaAs) ternary alloy, often regarded as GaAs and InAs binary alloys, is a III-V semiconductor compound with an increasing demand for the micro/opto -electronic industry [1,2]. Among the several processes used to growth InGaAs, the Magnetron Radio Frequency (RF) Sputtering offers a relative low cost fabrication technique. Understanding, controlling and characterizing the growing process is necessary in order to obtain the required film that best meets the specifications. Between the several characterization techniques, AM-AFM is a powerful, flexible and economic tool that enable us to study the surface and the heterostructure morphology [3,4]. Certainly with AM-AFM in phase contrast mode it is possible to study material properties [5]. Therefore, cross-sectional analysis in semiconductor heterostructures by AM-AFM allows us to study the inner features

[6]. On the other hand, μRAMAN spectra can give us the opportunity to study and understand interesting semiconductor materials properties, like crystal orientations, carrier concentrations and mixed crystals composition [10]. For all this, we are presenting our preliminary results of the GaAs/In/GaAs/In/GaAs/Si(100) heterostructure, grown by Magnetron RF Sputtering. Our interest to fabricate In/GaAs bilayers was to obtain the InGaAs ternary allow at the interface. Having the correct In content, a required band gap for solar cells applications can be obtained. For this reason, three samples were fabricated with different In content. The surface morphology was described in terms of the statistical quantities (Maximum Height value and RMS roughness) and Grain analysis, by using the watershed algorithm [4] (Grain Population Density, Mean Grain Size and Total Grain Volume). The hetero-structure features were studied by cross-sectional imaging, obtaining the topography and the phase images. Then, the μRAMAN spectra were obtained on the same area by using 532 nm and 785 nm laser lines respectively, detecting the phonon compositional related modes to GaAs and InAs.

[2] Experimental Procedure

High purity (95.5 %) GaAs(100) and In targets where used. The Si(100) surfaces were firstly degreased with acetone and methanol. After that, they were cleaned with hydrofluoric acid at 2 %, rinsed with deionized water, dried with nitrogen and introduced into the sputtering chamber. The growth chamber was evacuated to 1.2 x 10^{-6} Torr, then back-filled with high purity Argon gas to 1.2 x 10^{-3} Torr. For each sample, with the temperature of the Si(100) substrate at 580 °C, a GaAs buffer layer was deposited for 60 minutes, then the heterostructures were prepared as follows: with a temperature of 300°C , In deposition at 5, 10 and 15 min for Sample 1 (S1), Sample 2 (S2) and Sample 3 (S3), respectively, was performed. Then, another GaAs layer was deposited during 20 minutes at 580 °C. This process is then repeated once again for each sample, in order to deposit a total of two In-GaAs films onto a GaAs buffer layer with different In content. The AM-AFM and μRAMAN characterizations where performed at ambient conditions with the N8 NEOS SENTERRA system from Bruker. This equipment combines an optics SENTERRA Raman spectrometer with the Nano's N8 NEOS AFM, to allow the morphological, structural and chemical analysis of the same area. The cantilevers used were PPP-NCLR from Nanosensors, with 190 Khz resonance frequencies, and force constants around 48 N/m. The AFM was setup to work in AM-AFM contrast phase mode, with 195 nm Free oscillation Amplitudes (FA), Set Points (SP) of 50 %, 65 % and 80 %, Oscillation Frequencies (fosc) around 169 Khz, Scanning Speeds (SS) of 0.7 l/s, 0.5 l/s and 0.2 l/s, and the Free oscillation Phase (FP) was 90° for the cantilever free oscillation. The software used to treat the AFM images were Gwyddion [7] and NanoScope Analysis [8]. μRAMAN spectra were obtained with, a green laser (532 nm at 20 mW, 350 nm spot diameter), and a red one, (785 nm at 100 mW and 550 nm spot diameter).

[3] Results and Discussions

After optical imaging inspection, it was selected an area for each of the samples, from where μRAMAN and large scales AFM images were obtained. Then, 10 x10 μm^2 areas were imaged (figure 1) to perform the grain discretization by the Gwyddion watershed algorithm [4]. The statistical quantities and grain statistics resulted are presented in table 1. The cross-section topographic and phase images, figures 2(a,g), 2(b,h) and 2(c,i), for S1, S2 and S3, respectively, are presented with their height and phase profiles. At this particular locality they presented a film thickness of about 2.16 μm for S1, 3.85 μm for S2, and 6.15 μm for S3. Note that these values are slightly larger than those observed by the Maximum heights values presented in table 1. The phase signal presented a different behavior when the tip was interacting with the Si surface (left part of the image), with the film (middle and/or right), and with the free space, after the specimen presence (right part).

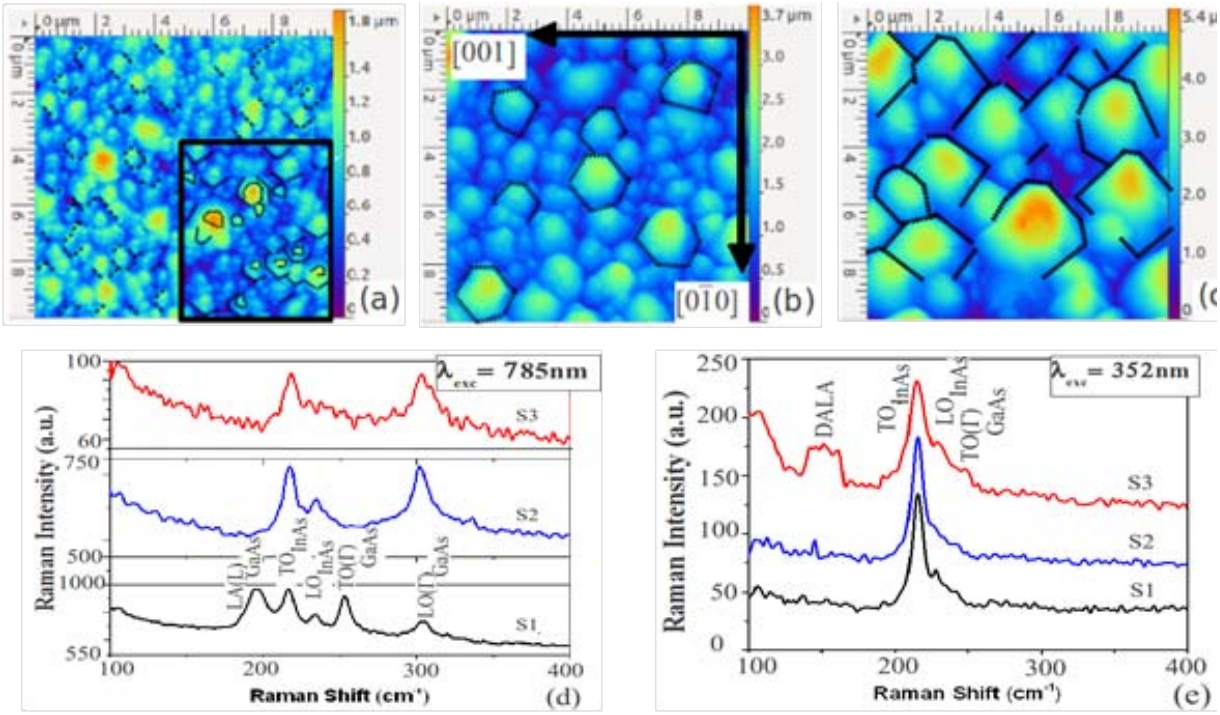

Figure 1: AFM topography images for S1, (a), SP = 50 % and SS = 0.5 l/s, The inset is an amplification of the zone below. For S2, (b), SP = 65 % and, SS = 0.6 l/s. And for S3, (c), SP = 50 %, and SS = 0.5 l/s. These images presented linear features (marked with black lines). The corresponding μRAMAN for all the samples are resumed in figures (d) and (e), for the 785 nm and 532 nm laser lines respectively.

Table 1. Statistical quantities and Grain statistics obtained from the AFM images of figure 1.

	Statistical Quantities		***Grain Statistics***		
Sample	**Maximum height [μm]**	**RMS Roughness [μm]**	**Grain population density [μm^{-2}]**	**Mean grain size [μm]**	**Total grain volume [μm^3]**
1	1.79	0.24	11.2	0.18	48
2	3.78	0.51	1.75	0.56	121
3	5.44	1.01	0.73	0.82	227

From figure 1(d), all the μRAMAM presented the two-phonon modes behaviour of first-order Raman scattering, TO and LO, of both InAs and GaAs. These spectra presented a shift of 5 cm^{-1} to lower frequencies with respect to the bulk, we are considering that these shifts are caused by the In content, due to the change of the integrated intensity, and the FWHM observed in InAs and GaAs Raman peaks. The average residual strain in the polycrystalline samples disappeared, because it is randomly distributed over the samples and then relaxed at the grain boundaries [9]. From cross-sectional SEM images (not presented here) we did not observe cracking in our samples. Additionally to the InAs and GaAs LO and TO phonon modes, the LA(A) mode centered at 195cm^{-1} is evident for S1. When the In-GaAs/In layers were excited with the 532 nm laser, figure 1(e), the GaAs-like LO phonon mode is not evident any more. However a weak signal of TO GaAs-mode can be identified, as well as the strong signal centered at 215 cm^{-1}, corresponding to a InAs TO phonon mode, with two small shoulders to the right, related to LO InAs and TO GaAs phonon modes. Finally, the band presented at 150 cm^{-1} (possibly produced by a Disorder Activated Longitudinal Acoustic (DALA) effect) increased in intensity while the In layer thickness increased.

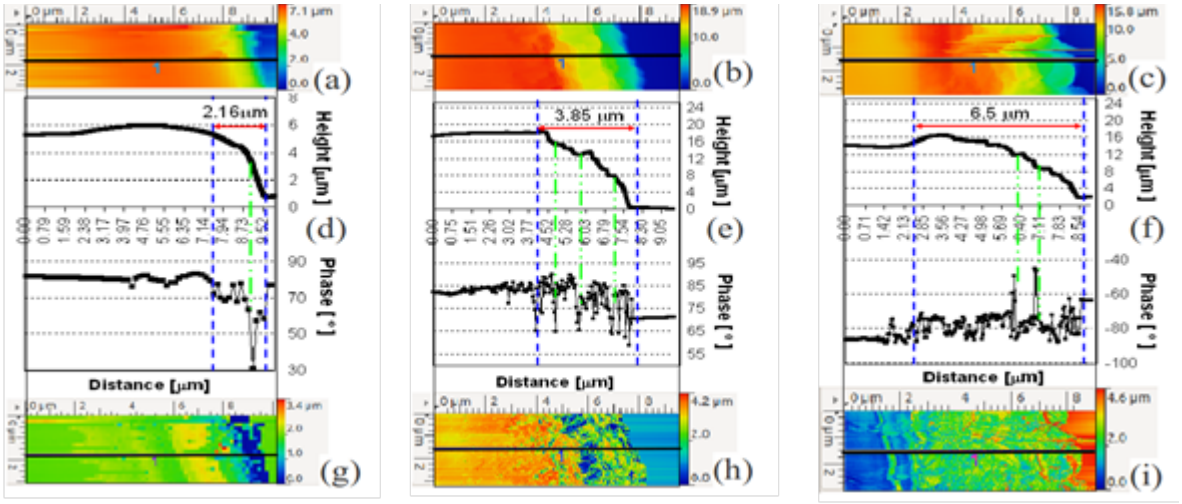

Figure 2: AFM Topography and Phase cross-sectional qualitative comparative analysis.

In summary, grain lateral dimensions and heights were increased upon larger In deposition times. Linear features with some order in certain crystallographic orientations were formed. A topography V.S. phase qualitative analysis of the specimens cross section, presented different phases values between the Si(100), the GaAs/In/GaAs/In/GaAs films and the free space. While the topography profile decreased logarithmically, the tendency of the phase signal did not present this behavior for all the samples. Raman spectra and composition analysis, presented the phonon related modes to GaAs and InAs. This Mean that the ending semiconductor compound was presented in the fabricated heterostructures. However, for the moment it is very premature to give a conclusion regarding the correct In content for a suitable InGaAs ternary alloy at the interface.

[4] Acknowledgements
We appreciate the “Instituto de Ciencia y Tecnología del Distrito Federal, (IcyTDF)” now “Secretaría de Ciencia, Tecnología e Innovación del Distrito Federal” (SECITI), for financial support. Sincere gratitude to Prof. José Trujillo and M.S. Marving Soriano. R. Bernal appreciate very much to “Colciencias – Colombia” for the financial support (“Francisco José de Caldas -2011” scholarships).

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