Multilayer growth of $Al_xGa_{1-x}As$/GaAs thin films using an elemental-arsenic-based-MOCVD system

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Abstract: GaAs and its alloys have proved to be successful semiconductor materials for the optoelectronic device industry, unfortunately its production by MOCVD systems require the use of a highly toxic compound as is the arsine. In this work we report some results of the studies related to the growth and characterization of $Al_xGa_{1-x}As$ /GaAs/ $Al_xGa_{1-x}As$ epitaxial layers obtained by Metal Organic Chemical Vapor Deposition (MOCVD) using solid arsenic instead arsine. The main goal is to explore the ability of this system to grow multilayer structures like quantum wells. The use of metallic arsenic could introduce important differences in the growth process due to the absence of the hydride group V precursor (AsH_3), which is manifested in the electrical and optical characteristics of both GaAs and $Al_xGa_{1-x}As$ layers. The gallium and aluminum precursors were trimethylgallium (TMGa) and trimethylaluminum (TMAl), respectively. The arsenic was introduced by diffusion on the growth zone using a metallic arsenic source. The most serious difficulty on growing $Al_xGa_{1-x}As$, is the incorporation of large amount of impurities as carbon and oxygen. The characterization of these epilayers was performed using Hall effect, low-temperature photoluminescence, Raman spectroscopy, Atomic Force Microscopy and Secondary Ion Mass Spectroscopy (SIMS).

Keywords—Elemental arsenic; MOCVD AlGaAs/GaAs epilayers; photoluminescence (PL); Atomic Force surface morphology; secondary ion mass spectroscopy (SIMS)

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I. INTRODUCTION

AMONG the III-V semiconductor materials, the $Al_xGa_{1-x}As$/GaAs/$Al_xGa_{1-x}As$ epitaxial layers have been extensively studied due to their wide range of characteristics and advantages for the elaboration of optoelectronic devices. The fabrication of modern electronic devices requires the growth of epitaxial layers with the purity and crystalline perfection demanded by its optical applications. The crystalline quality of the epitaxial layers strongly affects the device characteristics such as radiation efficiency and electrical properties, although this is not the only one that dominate the performance of these devices, so it is important not only obtaining the high quality material but also obtaining it with good quality of interfaces between the epitaxial layers, and with the minimum of roughness. Characteristics like the abruptness or flatness, and a minimum number of

interface defects are the optimal ones that must be reached, in order to obtain sharp transitions in the photoluminescence radiation and in general, to obtain the wide range of optical characteristic of the multilayer structures as single quantum wells and multiple quantum well structures [1]. Features as thickness control, the electrical and optical properties and crystal quality of the thin films are tightly associated with the growth technique used for its preparation. The metal organic chemical vapour deposition (MOCVD) system has been widely used for the growth of epitaxial semiconductors layers, with high quality and excellent surface morphology, however, this technique has as main problem the use of highly toxic gases, as is the case with the use of arsine and phosphine, both chemical compounds are used as precursors of the III-V semiconductor compounds. By other side, the use of nonconventional MOCVD systems, in which the arsine has been substituted by solid arsenic is cheaper and safer option [2,3]. In this work we present the results obtained using one of these growth systems, in which the use of arsine has been substituted by elemental arsenic. Solid arsenic is safer due to its low vapour pressure at room temperature and it is easy to store.
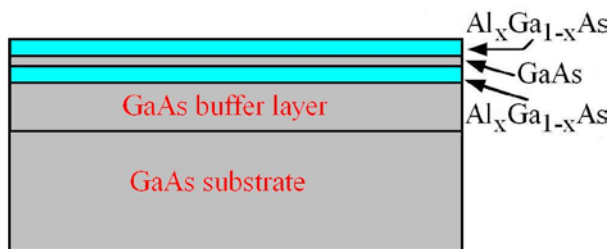

Fig. 1. Sample structure

Most of the grown $Al_xGa_{1-x}As$ layers have the same aluminum molar fraction (x=0.3 average).

II. EXPERIMENTAL DETAILS

All the layers presented in this work were grown using electronic grade trimethylgallium (TMG) and trimethylaluminium (TMA) as gallium and aluminium precursors, respectively, and metallic arsenic (9N) as the arsenic source. All samples were grown at atmospheric pressure by a horizontal modified MOCVD growth system. The MOCVD system consists of a horizontal quartz tube operating at atmospheric pressure. The substrates were placed on a graphite substrate holder and heated by a set of infrared lamps. The arsenic supply was controlled by a second independent furnace. The III/V ratio used in the growths can be easily adjusted by controlling the arsenic source temperature or/and controlling the

hydrogen flow through the metallorganic vessels by means of electronic mass flow controllers (MFC). The growth atmosphere consists of palladium purified hydrogen (H_2). The main characteristics of the MOCVD reactor were reported elsewhere [4]. The substrate preparation involves, as usually, degreasing by organic solvents, surface oxide elimination by HCl and a surface chemical etching using $H_2SO_4:H_2O_2:H_2O$ (5:1:1). As a last step, the substrates were rinsed in deionized water, and the GaAs wafers were dried by nitrogen blowing to eliminate any water trace. The first grown structures were epitaxial layer structures consisting of the following layers: 1) an undoped GaAs buffer layer, 2) a second $Al_xGa_{1-x}As$ layer, 3) a very thin GaAs layer and 4) an $Al_xGa_{1-x}As$ as a barrier. The growth of the structures is finished growing a very thin layer of GaAs as a protective layer. In some other cases following the same sequence, the GaAs layers were grown between the cladding $Al_xGa_{1-x}As$ layers. The thicknesses of these layers were in decreasing order toward the surface, as is illustrated in Fig. 1

For the low-temperature photoluminescence (LT-PL) measurements the samples were mounted on a cold finger of a closed-cycled liquid helium cryostat. The 632.8 nm line of a helium-neon laser was used as over gap excitation with a nominal power density of 20 mW/cm^2. Emitted radiation was detected with an GaAs:Cs photomultiplier R636 Hamamatsu and analyzed through an Anton monochromator. For studying the $Al_xGa_{1-x}As$ layers composition and their homogeneity Raman spectroscopy measurements were performed on the grown samples. Raman scattering experiments were carried out at room temperature using the 5145 Å line of an Ar^+-ion laser at normal incidence for excitation. The light was focused into a spot of 6.0 µm diameter on the sample using a 50x (numerical aperture 0.9) microscope objective. The laser power used in these measurements was 20 mW. Care was exercised to not heat the sample inadvertently to the point of changing its Raman spectrum. Scattered light was analysed using a Jobin-Yvon T64000 triple spectrometer, operating in the subtractive configuration, and a multichannel charge coupled device detector cooled at 140 K using liquid nitrogen. Typical spectrum acquisition time was limited to 60 sec to minimize the sample heating effects mentioned above. Absolute spectral feature position calibration to better than 0.5 cm^{-1} was performed using the observed position of silicon, which is shifted by 521.2 cm^{-1} from the excitation line. The electrical properties of the layers were studied using Hall Effect by the Van der Pauw method at 77 and 300 K. The investigated samples were square shaped with dimensions of 5.0 mm × 5.0 mm and thickness of 0.48-2.97 µm. The samples were

provided with four ohmic contacts by alloying small balls of indium on the sample corners, at 400°C for 1.0 minute in a nitrogen atmosphere. The linearity and symmetry of the ohmic contacts were tested following the procedure described in the ASTM standards [5].

III. EXPERIMENTAL RESULTS AND DISCUSSION

After the substrate preparation, the first grown layer was a GaAs buffer layer with a thickness of approximately 0.2 µm and in some cases 0.5 µm. The substrate temperatures used for the growth of the GaAs buffer layer ranged from 600 to 875°C with an arsenic flux of 7.0 µmol/min. This arsenic flux corresponds at 560°C for the arsenic source, in the growth system. The buffer layer surface morphology was evaluated using Atomic Force Microscopy (AFM). All the examined GaAs buffer layers presented a like-mirror aspect. Figure 2 shows the typical surface morphology of the buffer layers.
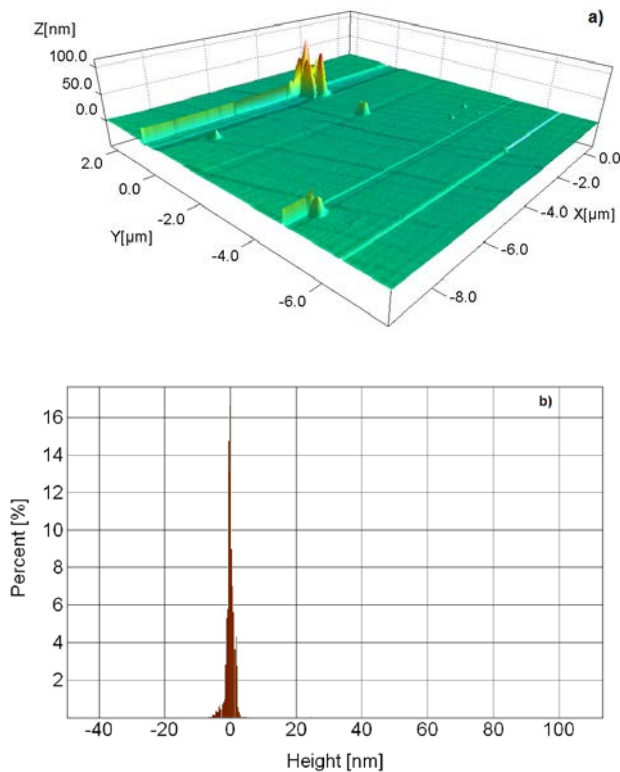

Fig. 2. a) Surface Morphology of the GaAs buffer layer obtained by Atomic Force Microscopy, which was grown at 750°C. b) Histogram of heights distribution of the GaAs layer,

As it can be seen in Fig. 2a the roughness of the layer is not larger than 10 nm. The growth temperature of the shown sample was ~750°C. In Fig. 2b is shown the height distribution. These results contrast with the

obtained ones in the case of the ternary alloy, as is presented in Fig. 3, the AFM scans shown that the samples grown with this growth system presented a large roughness. In the case of the sample presented, as can be observed in the vertical scale, the peak value is about 60 nm. Although growth parameters were varied, the degree of surface roughness did not decrease, whereby it is thought that this degree of roughness is related with the surface growth kinetic on the substrate.
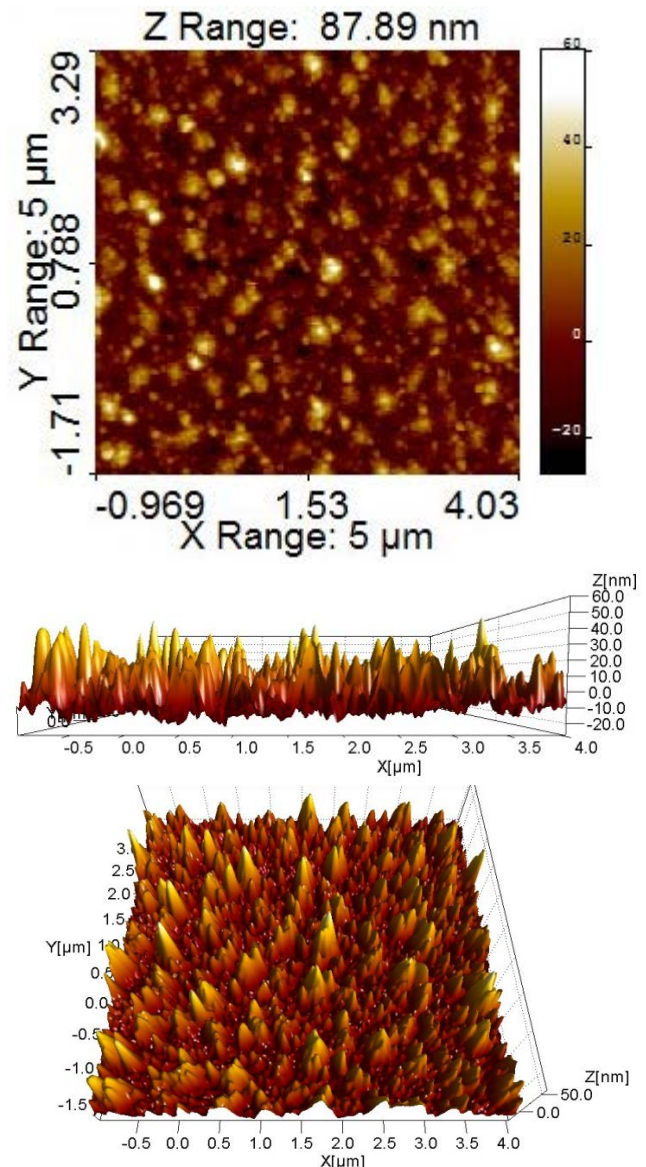

Fig. 3. An $Al_xGa_{1-x}As$ typical surface scanning by AFM, x=0.3.

This degree of roughness is a serious limitation if the goal is the growth of multilayer structures based in quantum effects like the exciton confinement and radiation; this due to the size of the confinement layer is

out of precise control with this growth system, producing a weakness and wide photoluminescence signal cause of the large width distribution.

In Fig. 3 is illustrated the degree of roughness obtained for $Al_xGa_{1-x}As$ samples grown with solid arsenic. For growth temperature higher than 800°C the surface appeared hazy and in some cases eventually becomes polycrystalline as was examined by X ray measurements [6]. The $Al_xGa_{1-x}As$ layers were grown in a wide interval of aluminium molar concentration, but in all cases the roughness was high.
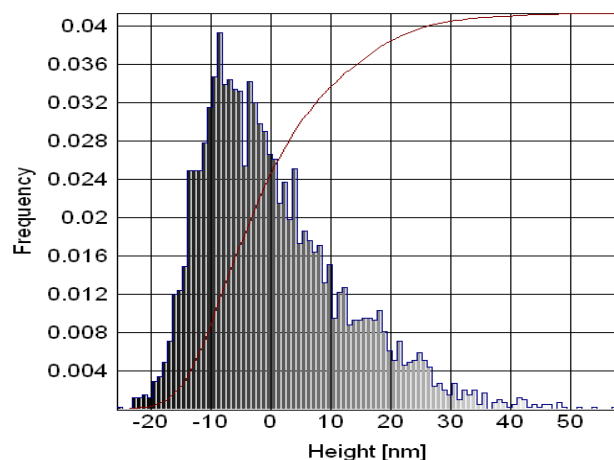

Fig. 4. Histogram of heights distribution.

The way in which the heights are distributed on the layer surface can be appreciated in Fig. 4. This histogram has been obtained from 100 points on the surface. The more common explanation for the high roughness degree in the $Al_xGa_{1-x}As$ layers is the very low surface mobility of aluminium during the growth process. Furthermore, in the growth system in which the AsH_3 has been replaced by elemental arsenic the growth kinetic has been drastically modified [7, 8]. As has been pointed out, the substrate temperatures used for growing the GaAs buffer layer ranged from 600 to 875°C and the arsenic temperature was 560°C. In this range, the free carrier concentration increases as the growth temperature is increased. The electrical characterization results for $Al_xGa_{1-x}As$ layers are summarized in the Table I, at 650°C the electron concentration was approximately 7×10^{15} cm^{-3}, while for samples grown at ~870°C the carrier concentration was approximately 2×10^{17} cm^{-3}, the 300 K mobility of these layers was approximately 2000 cm^2/V·s. In some cases, the 77 K mobility could not be measured due to the impossibility of obtaining good ohmic contacts at this temperature. As the growth temperature was increased the carrier concentration only registered a slight increase. The Hall

effect measurements at 77 K show that the grown samples are highly compensated. Several authors have reported that carbon concentration increases in a linear proportion as the growth temperature is increased. The absolute carbon concentration in $Al_xGa_{1-x}As$ epilayers could increase at levels larger than 10^{19} cm^{-3}. Although the layers were grown without intentional doping, nevertheless, it was found a large amount of incorporated residual impurities. These residual impurities can be considered as a result of the organometallics impurities and from the decomposition of these compounds, as have been found by Spectroscopy Ion Mass Secondary (SIMS). These residual impurities are mainly carbon, oxygen and, silicon which concentrations are about 10^{18}, 10^{21} and $10^{16} – 10^{17}$ cm^{-3}, respectively.

TABLE I. ELECTRIC MEASUREMENTS OBTAINED BY HALL EFFECT OF $Al_xGa_{1-x}As$ LAYERS GROWN BY THE ELEMENTAL−ARSENIC-BASED-MOCVD SYSTEM. THE TABLE CONTAINS SOME GROWTH PARAMETERS.

Sample	T_G (°C)	T_{As} (°C)	N_{300} (cm^{-3})	μ_{300} (cm^2/V·s)	Resistivity (Ω·cm)
1169	750	570	1.34×10^{17}	538	0.09
258	750	570	2.33×10^{16}	876	0.03
1235	800	570	2.20×10^{17}	943	0.02
126	820	585	1.40×10^{16}	1718	0.03
1173	850	570	4.20×10^{17}	1272	0.01
57	870	570	1.70×10^{16}	2290	0.06
Q39	875	565	7.30×10^{16}	1421	0.06

From the experimental results, we may note some important facts. The electron mobility at 300 K for the $Al_xGa_{1-x}As$ epilayers grown with metallic arsenic is comparable to the mobility of the samples grown in arsine-based MOCVD systems [7-9]. The differences between the measured carrier concentrations with those taken from the reference [10] can be related to the presence of carbon, silicon and oxygen as residual impurities. As several authors have shown, the oxygen concentration in MOCVD $Al_xGa_{1-x}As$ samples lies in the range of 10^{17} to 10^{19} cm^{-3}. Otherwise, as our SIMS measurements shown, the carbon concentration can be as high as 10^{19} cm^{-3}. With reference to the role of the arsenic source in the epitaxial layer characteristics, the changes on the arsenic pressure apparently do not influence the electrical properties of the samples. As can be seen in Table I, for the samples Q39 and 126, with different growth conditions, the mobility and the carrier concentration have similar magnitudes. In fact, the room temperature mobility for all the samples varies in the range 1000 to 2500 cm^2/V·s, these values are in

the range of those reported by other authors for arsine-based-MOCVD process.

Figure 5 illustrates the 12 K photoluminescence spectra of the GaAs buffer layers, which present two dominant radiative bands that are situated in the excitonic and of impurities regions. In Fig. 5a, the peaks labelled by **a** and **b** correspond to transitions produced by the carbon as acceptor impurity, the **a** corresponds to the radiative transition conduction band to neutral acceptor at 829.4 nm (e-A°), and the **b** peak to the radiative transition neutral donor to neutral acceptor at 831.6 nm (D°-A°). The existence of the free exciton peak in the LT-PL spectrum demonstrates the good crystalline quality of the GaAs layers [12]. In Fig. 5b, the first one is constituted by three bands that are associated at free exciton (F, X) (817.9 nm) [7, 11], the second, exciton state bound to neutral donor, $(D°,X)_{exc}$ (818.7-818.8 nm), the third corresponds at $J=1/2$ exciton bound to neutral acceptor, $(A°,X)_{1=2}$ (819.7 nm).
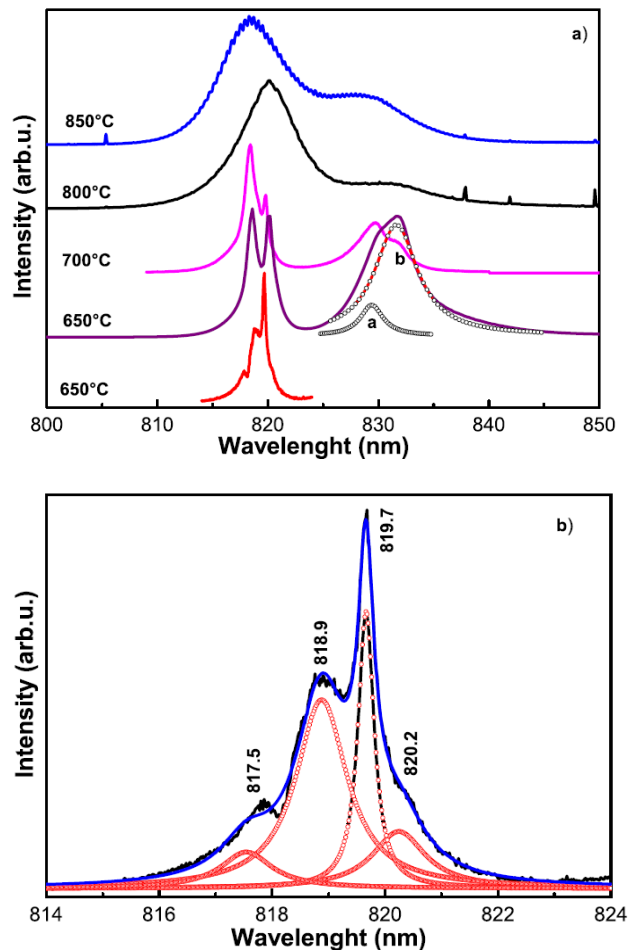

Fig. 5. GaAs buffer layers photoluminescence spectra for different growth temperatures.

In the case of the $Al_xGa_{1-x}As$ samples the photoluminescence measurements showed that the samples grown using temperatures below 800°C did not display appreciable photoluminescence signal. In order to obtain photoluminescence signal from the $Al_xGa_{1-x}As$ layers was necessary to grow them at temperatures higher than 820°C, which are high in comparison with the temperatures required in other growth systems as in the same arsine-based-MOCVD. Fig. 6 shows the LT-PL spectra of AlGaAs sample grown at 850°C, the weaknesses and in some cases the lack of photoluminescence signal is associated to the great quantity of residual impurities that come from the metallorganic precursors [7]. It is widely accepted that oxygen is a residual impurity that introduces deep traps into $Al_xGa_{1-x}As$ [12-14], In addition, when the growth temperature is increased the traps concentration is reduced. A common characteristic of $Al_xGa_{1-x}As$ layers is the large "full width at half-maximum" (FWHM) of the peaks corresponding to the $Al_xGa_{1-x}As$ excitonic and the acceptor-donor bands. Typical values of the FWHMs of the epilayers were around 80 meV, which are associated with the incorporation of residual impurities and to the variation of aluminium molar concentration due to mass controllers. The wide and shape of this peak make difficult to evaluate the aluminium molar concentration from the BE-peak position in the low-temperature photoluminescence spectrum.
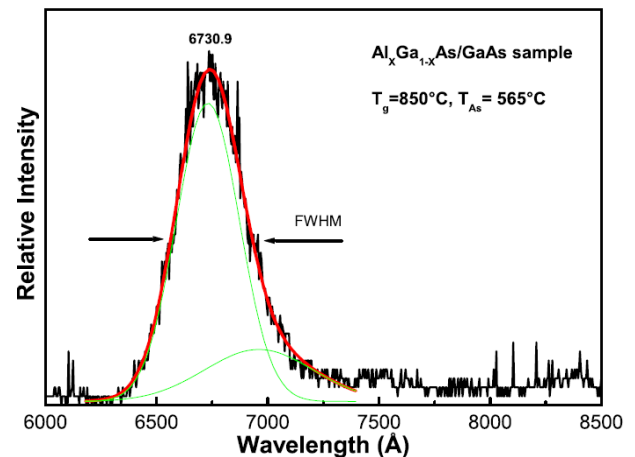

Fig. 6. Photoluminescence spectra measured at 12K of $Al_xGa_{1-x}As$ layers.

Figure 7 shows the typical Raman spectrum of the $Al_xGa_{1-x}As$ samples grown by nonconventional MOCVD. The Raman spectrum presents five vibrational bands centred at 278, 377, 552, 650 and 746 cm^{-1}. The dominant bands can be associated to the GaAs, and AlAs vibrational modes. A standard fitting

procedure performed in the interval of 215–450 cm^{-1} suggests this association. Such bands can be deconvoluted in four Lorentzian line shape signals as shown in Fig. 7, which are located at 268, 277, 366, and 378 cm^{-1}.

Taking into account that phonons are active in the first-order Raman process in backscattering on the (001) face, one may assign our vibrational bands to the TO-GaAs-like (268 cm^{-1}), LO-GaAs-like (277 cm^{-1}), TO-AlAs-like (366 cm^{-1}) and LO-AlAs-like (378 cm^{-1}). The low frequency asymmetry of the GaAs-like mode is then obviously due to the contribution of the scattering process of phonons with nonzero **q** vectors that become active due to the alloying disorder process. The TO-GaAs mode, which in principle is forbidden in the experimental geometry for the (100) orientation of the substrate, becomes active by the breakdown of the selection rules in the backscattering configuration. This breakdown is attributed to structural defects in the alloy originated from compositional fluctuations, elastic scattering and by the ionized doping impurities. Its appearance indicates that the crystalline quality of the $Al_xGa_{1-x}As$ layers is not perfect but in general the crystalline quality is fairly good as has been demonstrated. The modes LO-GaAs like, TO-AlAs like and LO-AlAs like, respectively, are originated on the ternary alloy. Due to the thickness of the $Al_xGa_{1-x}As$ epilayers, the modes corresponding to GaAs should not be assigned to the buffer layer. A possible explanation of the presence of the GaAs signals is the possible existence of crystalline defects in the epitaxial.
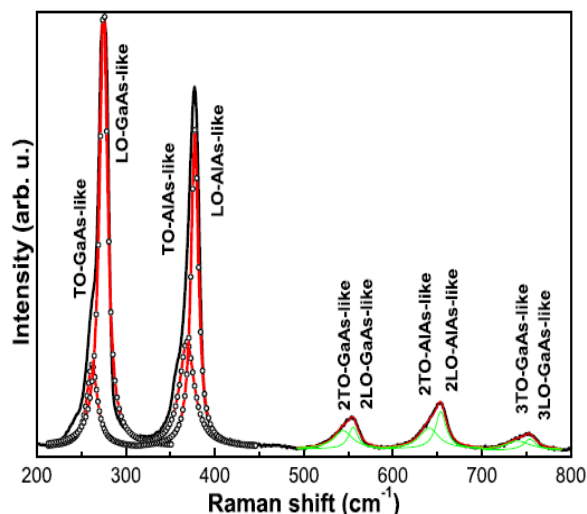

Fig. 7. Typical Raman spectrum of the $Al_xGa_{1-x}As$ sample grown by elemental-arsenic-based-MOCVD system.

The chemical composition of the $Al_xGa_{1-x}As$ layers was studied by secondary ion mass spectroscopy. Fig. 8 illustrates the SIMS concentration profiles for the residual impurities detected in one of the typical $Al_xGa_{1-x}As$ layers. The main impurities are silicon, carbon and oxygen. In the region corresponding to the $Al_xGa_{1-x}As$ layers, the silicon concentration approximately acquires a constant level that can be observed in figure but in the region of the GaAs buffer layer the silicon concentration reduces until the SIMS detection limit for silicon. In the $Al_xGa_{1-x}As$ layers, the silicon concentration slightly reaches a greater concentration to $1\times10^{17}cm^{-3}$.
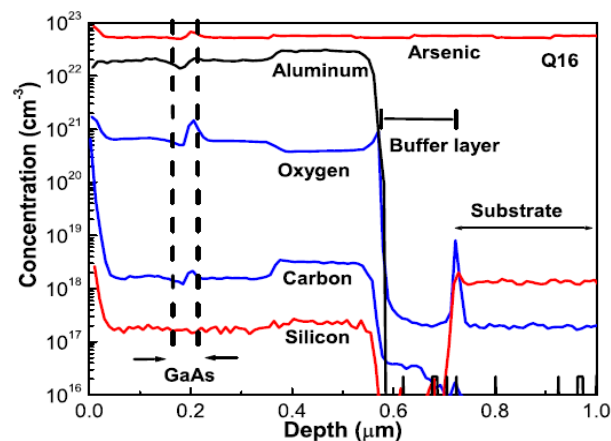

Fig. 8. Profile of concentration of residual impurities in the sample Q16 measured by SIMS. The variations of aluminium concentration during the growth do not result in the increase of oxygen.

According to the profiles observed in the samples, the source of silicon contamination is the aluminium precursor (TMAl). From the Hall effect measurements and taking into account the silicon behaviour like donor impurity, it is possible to be concluded that the silicon is the one that determines the type of conductivity in the layers. Although, the silicon is of group IV and could also act like acceptor impurity, in the growth of $Al_xGa_{1-x}As$ in vapour phase. It has been demonstrated that it introduces a single donor level. The behaviour of oxygen for the samples presents an abrupt change in the limit region between the GaAs and $Al_xGa_{1-x}As$ layers. This change is related to the time at which the TMAl is introduced to the growth chamber to initiate the growth of the $Al_xGa_{1-x}As$ layer, in such a way that the oxygen presence is directly related to the aluminium source. The oxygen origin is related to the presence of alcohoxides like residual impurities in TMAl. As is observed in Fig. 8 for the oxygen case, the curve indicates a clear tendency to accumulate in the inverted

interface GaAs/Al$_x$Ga$_{1-x}$As, this result is very important since oxygen do not only produce non radiative recombination centres in the GaAs and Al$_x$Ga$_{1-x}$As layers, but also will affect the structural characteristics of the interface, deteriorating in consequence its optical and electrical properties.

In order to explore the capabilities of this growth system, for growing multilayer with thicknesses near 100 nm it was grown the sample Q23, which SIMS profile is showed in Fig. 9. In this sample the number of layers grown was 8: buffer layer (GaAs)- Al$_X$Ga$_{1-X}$As - GaAs - Al$_X$Ga$_{1-X}$As... All the AlGaAs layers were grown with the same molar aluminum concentration. The substrate temperature was 855°C and the arsenic source temperature was 570°C, with these temperatures the molar fluxes calculated were 9 µmol/min or the arsenic, 2.2×10^{-6} mol/min for the TMGa. And 2.4×10^{-6} mol/min for the TMAl. For purging the growth chamber after the grown of the inverted interface (Al$_X$Ga$_{1-X}$As/GaAs), it was practice interruption times of 10 minutes, this was done to assure the adequate purge of the growth chamber and the abruptness composition change in the interface. In the case of the sample of Fig. 9, it can be observe that the GaAs layers are not well defined, although it can be attributed to the large amount of roughness presented by the layers.
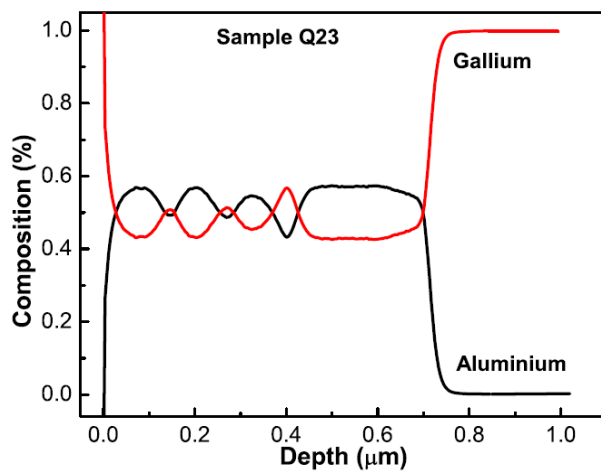

Fig 9. Depth profile for a multilayer sample

IV. CONCLUSION

In this work, we report the successful growth of Al$_X$Ga$_{1-X}$As/GaAs multilayer structure using a modified MOCVD growth system and solid arsenic as precursor. The GaAs and Al$_X$Ga$_{1-X}$As samples presented photoluminescence signal and a good homogeneity measured by Raman spectroscopy. In the experiments, a wide range of parameters were explored but the

samples, in general, showed a hazy surface and high roughness. These characteristics can be associated with the processes of decomposition, diffusion, incorporation, elimination of byproducts and in general to the modified growth kinetics, due to the use of solid arsenic.

We have studied Raman scattering of Al$_x$Ga$_{1-x}$As films grown by MOCVD at several growth temperatures from 650°C to 800ºC using solid arsenic as arsenic source. The best samples were grown at around 750ºC, for samples grown at lower or higher temperatures some forbidden Raman signals were observed. Those forbidden signals can be provoked by the resulting high concentration of the residual impurities.

The Al composition in the ternary alloy was determined using the peak frequency of the LO like modes. The small shoulder on the low-frequency side of the GaAs-like LO phonon is the GaAs-like TO mode, in principle forbidden in the experimental geometry, can be assigned to the high residual impurity of the AlGaAs layers. Its appearance suggests that the crystal quality of the AlGaAs layers grown by MOCVD using elemental arsenic is not perfect, but its small intensity indicates that the quality is good.

The samples grown by MOCVD using elemental arsenic were highly doped with oxygen, carbon and silicon. The origin of the impurities is related to the chemical grade of the organometallic sources. We obtained, by Hall effect, free carrier concentration about 1.7×10^{16} - 4.2×10^{17} cm^{-3}, but low mobility, meaning that the samples are highly compensated. These obtained results are compared with the reported ones in the literature for alternatives growth systems without arsine.

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