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Growth and characterization of $Al_{1-y}In_yAs/Ga_{1-x}In_xAs$ strained multiple quantum wells

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Highly strained $Al_{1-y}In_yAs/Ga_{1-x}In_xAs$ multiple quantum wells have been grown by atomic layer molecular beam epitaxy on [001]-GaAs substrates. Raman scattering and x-ray diffraction techniques have been used to determine layer composition and strain for a wide range of In concentration ($0 \leqslant x \leqslant 0.44$). When a thick buffer layer of AlInAs is used, commensurate growth of the quantum wells takes place if the In content of the barriers equals that of the buffer layer. Then the strain is only accumulated in the wells. The GaInAs wells can be either under biaxial tension or compression depending on the relative In content in wells and barriers.

I. INTRODUCTION

The possibility of modifying the characteristics of epitaxial layers by mechanical strain is at the origin of the development of devices based on strained pseudomorphic semiconductor layers of materials having different free lattice parameters. During pseudomorphic growth of epitaxial layers the substrate determines the lattice parameter in the plane perpendicular to the growth direction. The epitaxial layers then suffer a tetragonal elastic deformation that accumulates elastic energy in the sample. Above a critical thickness, misfit dislocations appear and the strain in the layers is relaxed.[1–5] In strained layer superlattices (SLSs) and multiple quantum wells (MQWs) two different critical thicknesses must be considered: the first one is related to the relaxation of individual layers, one relative to the other, whereas the second one is related to the relaxation of the superlattice considered as a whole, relative to the substrate.[6] When the substrate is identical to one of the superlattice constituents, the strain is mostly localized in the other one. In this way, the strain distribution between the two constituent materials can be governed, to some extent, by using a thick buffer layer of the proper composition between the substrate and the MQW or SL system. A good example of this is the growth of Ge/Si SLSs on Ge_xSi_{1-x} buffer layers.[7,8]

Semiconductor strained layers have been shown to enhance the ultimate performance of devices obtained with lattice matched material, as long as the layers are thin enough to avoid misfit dislocations. In III–V compound SLSs and MQWs the smaller band-gap material can be either under in-plane biaxial tension (as for example in the $Al_{1-y}In_yAs/GaAs$ system) or compression (as in $Ga_{1-x}In_xAs/GaAs$ heterostructures). Generally, for a given substrate and system, only one of the two strained configurations can be achieved. This restriction does not hold for $Al_{1-y}In_yAs/Ga_{1-x}In_xAs$ SLSs and MQWs, and the versatility of these structures makes them very attractive for lasers, modulators,[9–11] and other devices based on nonlinear optical properties.[12–14]

In order to assess the full potential usefulness of a given strained layer system it is necessary to determine accurately the built-in strain and how it affects the critical thickness of that system. Raman spectroscopy has been successfully used to measure strain in diamond and zinc-blende type semiconductors.[15,16] The linear dependence of the optical phonon frequency on strain of these materials provides a rapid, precise, nondestructive, and spatially resolved technique to determine the internal strain in the different constituents of a strained layer system. Strain determination by means of Raman scattering has been reported for a variety of group IV,[7,8] and III–V[17–26] pseudomorphic layered compounds. Olego *et al.*[27] have been able to determine strain profiles versus depth in ZnSe single epitaxial layers grown on GaAs and in $ZnSe/ZnS_xSe_{1-x}$ SLSs by measuring Raman spectra for different laser wavelengths (i.e., penetration depths). Venkateswaran *et al.*[28] have recently reported areal strain maps with 10 μm lateral resolution recorded by Raman scattering in [111]- and [001]-oriented $Ga_{1-x}In_xAs$/GaAs SLSs.

In this paper we report on the growth and structural characterization (composition, layer thicknesses, and strain) of

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$Al_{0.9}In_{0.1}As/Ga_{1-x}In_xAS$ MQWs for a wide range of compositions ($0 \leqslant x \leqslant 0.44$) by means of x-ray diffraction and Raman scattering. We find that positive or negative strains up to 3% can be elastically accommodated in the quantum wells if they are grown on a thick relaxed buffer layer with the same-composition as the barriers. These results can be of relevance for the design of GaInAs-based strained-layer devices.

II. EXPERIMENTAL

The samples used in this work were grown by atomic layer molecular beam epitaxy (ALMBE) on [001] GaAs semi-insulating substrates. After oxide removal, a 250-nm-thick GaAs buffer layer was grown by conventional MBE with a substrate temperature $T_s = 585$ °C. The layer structure was grown at $T_s = 400$ °C by ALMBE in As_4-pulsed operation mode.[29] For the MQW structures, a 1-μm-thick nominally $Al_{0.9}In_{0.1}As$ buffer layer was grown in order to decouple the MQW system from the GaAs substrate. The lattice mismatch between $Al_{0.9}In_{0.1}As$ and GaAs is $f = 0.83\%$, which corresponds to a predicted critical thickness of $h_c < 20$ nm[4,30] or $h_c \approx 200$ nm,[5] depending on the authors. The thickness of the buffer layer was chosen far above any of these values trying to ensure a complete lattice relaxation. On top of the buffer layer, the MQW structure was formed by eight periods of $Ga_{1-x}In_xAs$ wells of thickness L_w, separated by 10-nm-thick $Al_{0.9}In_{0.1}As$ barriers (same composition as the buffer layer). A 2-nm-thick $Ga_{1-x}In_xAs$ cap layer was grown on top of the MQWs. Two samples with $L_w = 32$ and 40 Å well widths, respectively, were grown for different values of In concentration in the $Ga_{1-x}In_xAs$ layers, which has been varied from $x = 0$ to $x = 0.44$. The corresponding values of the lattice mismatch relative to $Al_{0.9}In_{0.1}As$ are $f = -0.82\%$ and $f = +2.38\%$, respectively. Right after the growth of each MQW pair of samples with a given x value, a third sample consisting of a thick $Ga_{1-x}In_xAs$ layer was grown on GaAs under the same growth conditions. The thickness of these layers (>1 μm) is large enough to ensure strain relaxation, so that they can be used as control samples for the Raman scattering measurements. In this way one can distinguish between the phonon frequency shift due to composition changes from the shift due to strain. Special care has been taken to grow every sample series (MQWs and control thick film) under the same growth conditions and within the same growth run. A check of the growth rates for GaAs, AlAs, and GaInAs was carried out prior to every growth run by reflection high-energy electron diffraction oscillations, to ensure that: (i) the composition (and the lattice constant) of the barrier layers is the same as in the AlInAs buffer layer and, (ii) the composition of the QWs is the same as that of the corresponding control samples, so that the different phonon frequencies are only due to the strain present in the wells.

The actual composition of the layers was derived from x-ray diffractometry (XRD) measurements. Double crystal x-ray diffraction (DCXRD) analysis was carried out by measuring rocking curves around the GaAs substrate reflection, in a high resolution DCXR diffractometer using an asymmetric [001] Ge crystal in the [004] reflection as monochromator.[31] Scans were recorded around symmetric

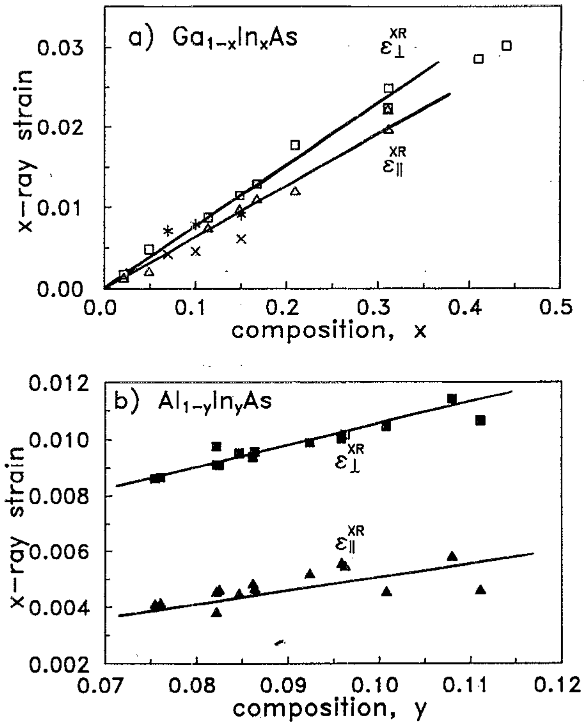

FIG. 1. Perpendicular (squares) and parallel (triangles) strain as a function of the In concentration obtained from x-ray measurements for (a) the $Ga_{1-x}In_xAs$ control samples, and (b) the $Al_{1-y}In_yAs$ buffer layers of the MQW structures. Data reported in Ref. 21 for $\epsilon_\perp^{xr}$ (*) and $\epsilon_\parallel^{xr}$ (X) are also shown in the figure. The lines are only a guide to the eyes.

[004] and asymmetric [511] GaAs reflections, each of them measured at least in two opposite azimuths in order to counteract the effect of the tilt of the epitaxial layer in XRD patterns.[32] These measurements give the lattice constant along the growth axis, $a_\perp$, and in the growth plane, $a_\parallel$, of the thick layers (AlInAs buffers in the MQW structures and GaInAs thick films in the control samples). The actual In concentration was calculated from those values. The period of the MQWs was eventually checked for some samples by single crystal diffractometry in a θ–2θ configuration, and was in good agreement with the nominal values.

To perform the Raman measurements the samples were mounted on an optical cryostat and maintained at 77 K. The Raman spectra were measured in backscattering geometry. The 5145 Å line from an Ar^+ laser was used as the excitation source. The barrier material $Al_{0.9}In_{0.1}As$ is transparent at this wavelength. For Raman measurements this offers the practical advantage of long penetration depths, thus probing a larger scattering volume of the highly absorbing wells. The power of the incident beam was typically 50–70 mW. The scattered beam was analyzed by a Dilor XY spectrometer equiped with an intensified cooled Si-photodiode array detector with a spectral resolution of $\simeq 2$ cm^{-1}.

III. RESULTS AND DISCUSSION

Figure 1 shows the perpendicular, $\epsilon_\perp^{xr}$, and parallel, $\epsilon_\parallel^{xr}$, x-ray strain values given by

$$\epsilon_{\parallel,\perp}^{xr} = (a_{\parallel,\perp} - a_s)/a_s, \tag{1}$$

where a_s is the substrate lattice constant.

They are obtained from the DCXRD measurements and plotted versus the In content of each sample, both for $Ga_{1-x}In_xAs$, [Fig. 1(a)] and $Al_{1-y}In_yAs$ [Fig. 1(b)] layers. Although in our experimental configuration the parallel lattice constant measurement has an appreciable error margin, we observe that $\epsilon_{\parallel}^{xr}$ has values close to but systematically lower than $\epsilon_{\perp}^{xr}$. This fact indicates that some residual strain, for which the formation of misfit dislocations is not favorable anymore, is still present. The composition of the samples has been obtained according to Vegard's law taking into account the departure from ideal relaxation through the expression of the "cubic" or "relaxed" lattice parameter $a_0(x)$ given by

$$\epsilon_0=\frac{a_0(x)-a_s}{a_s}=\frac{c_{11}}{c_{11}+2c_{12}}\ \epsilon_{\perp}^{xr}+\frac{2c_{12}}{c_{11}+2c_{12}}\ \epsilon_{\parallel}^{xr}, \quad (2)$$

where c_{ij} are the elastic constants of the material, which are dependent on the composition of the alloy. They initially have been taken to be those corresponding to the In content obtained from the perpendicular strain (assuming the material fully relaxed) and subsequently corrected in further iterations. We include for comparison in Fig. 1(a) some data extracted from Ref. 21 corresponding to $Ga_{1-x}In_xAs$ layers of the same thickness as in our samples.

The relaxation of an epitaxial layer on a mismatched substrate strongly depends on the thickness of the layer, on the growth conditions, and the materials involved, through their elastic behavior and the complex influence of growth kinetics in the relaxation process. The degree of relaxation can be quantified by the ratio

$$R=\epsilon_{\parallel}^{xr}/\epsilon_0, \quad (3)$$

where ϵ_0, defined in Eq. (2) represents the relative difference between the relaxed lattice parameter of the epitaxial layer and that of the substrate. R is equal to zero for a perfectly strained epitaxial layer and equal to unity for a fully relaxed one.

In Fig. 2 we have plotted the calculated values of the difference

$$\Delta\epsilon=\epsilon_{\perp}^{xr}-\epsilon_{\parallel}^{xr}=\frac{c_{11}+2c_{12}}{c_{11}}\ (1-R)\epsilon_0 \quad (4)$$

for different values of R as a function of In concentration, for $Ga_{1-x}In_xAs$ [Fig. 2(a)] and $Al_{1-y}In_yAs$ [Fig. 2(b)], together with the experimental values obtained for $\Delta\epsilon$. We observe that for GaInAs thick films the average degree of relaxation is close to 95%, whereas in the case of AlInAs, the lattice relaxation is significantly lower ($R\approx 0.65$). This result is consistent with the higher elastic stiffness of AlAs as compared to GaAs.

For one particular sample, the tilt between the epitaxial layer and the substrate has been measured evaluating the azimuthal dependence of the angular distance between substrate and epitaxial peaks. The obtained value for the tilt angle is $\beta=0.02°$, for a mismatch value of $f=0.24\%$. This result is comparable to the lowest angle reported for a similar mismatch,[33] indicating that lattice relaxation has been achieved with a low density of threading dislocations.

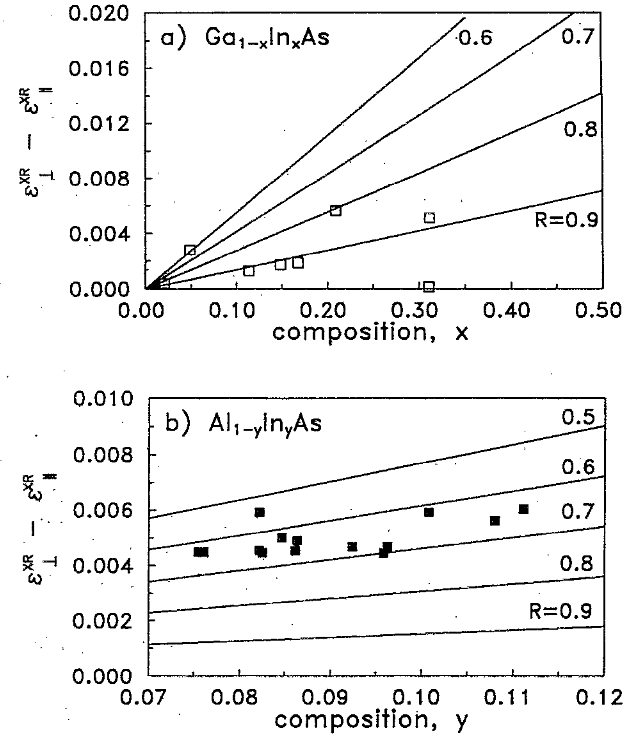

FIG. 2. Calculated strain differences for various degrees of relaxation R of an epitaxial layer grown on a GaAs substrate, as a function of In concentration. (a) $Ga_{1-x}In_xAs$ and (b) $Al_{1-y}In_yAs$. The experimental points are those corresponding to Fig. 1.

Figure 3 shows the Raman spectra of two $Ga_{1-x}In_xAs$ thick films (labeled TF) with different In content ($x=0$ and $x=0.36$) and the corresponding strained MQWs with nominally the same In concentration in the well layers. In these measurements both the incident and the scattered light are polarized along the [110] crystallographic direction. The crystal orientation allows the observation of Raman lines corresponding only to the LO phonons of the singlet type (i.e., vibrating along the growth axis).[15,22]

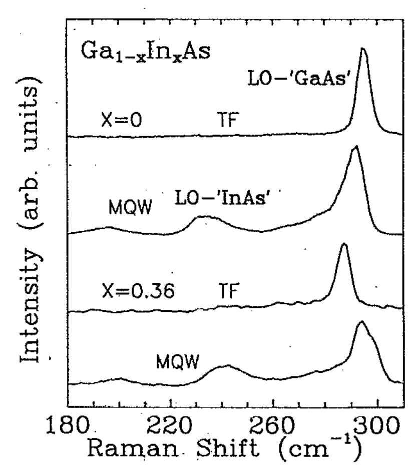

FIG. 3. Raman spectra of two $Ga_{1-x}In_xAs$ relaxed thick films with different In content and of two strained $Al_{1-y}In_yAs/Ga_{1-x}In_xAs$ MQWs (100 Å/32 Å) with the same In concentration in the well layers.

The Raman spectra of $Ga_{1-x}In_xAs$ as well as $Al_{1-y}In_yAs$ display a two-mode type behavior, i.e., two LO modes associated with the respective end compounds are present for the whole composition range.[33–36] The measured Raman spectra from the $Ga_{1-x}In_xAs$ thick films show only one peak at 290 and 285 cm^{-1}, respectively, that correspond to the GaAs-like LO-phonon mode. When comparing samples with the same In content, the GaAs-like peak appears shifted to higher energies with respect to the TF one in the MQW sample with $x=0.36$ and to lower energies in the sample with $x=0$. These shifts are directly related to the strain in the well and will allow its quantitative determination, as shown below. The peaks at $\simeq 237$ cm^{-1} correspond to InAs-like vibrations, which are present in both wells and barriers. We have performed a series of resonant Raman measurements in the energy range from 1.72 to 1.92 eV in order to elucidate the origin of the InAs-like vibrations observed in Fig. 3. The covered energy range corresponds to the $E_0+\Delta_0$ gap of $Ga_{1-x}In_xAs$ once confinement effects are taken into account. We observed indeed a clear resonant enhancement of the GaAs-like mode intensity, whereas the InAs-like mode remained unaltered. This indicates that the observed InAs-like mode originates in the barriers and therefore is not suitable to monitor the strain present in the well layers. The weak band at 190 cm^{-1} is due to disorder induced scattering by acoustic vibrations.[36]

The $Al_{0.9}In_{0.1}As$ barriers appear to be free of strain, as the AlAs-like LO-phonon frequency (not shown) measured in all our samples remains constant independently of well thickness and composition. As it has been shown previously by the DCXRD measurements the $Ga_{1-x}In_xAs$ thick films are almost completely relaxed [Figs. 1(a) and 2(a)]. We assume therefore that the frequency shift of the GaAs-like LO-phonon mode in the MQWs compared to that of the thick films is induced by the lattice mismatch strain in the $Ga_{1-x}In_xAs$ well layers. Therefore, this mode will be utilized to evaluate the strain.

As can be seen in Fig. 3, compressive strain appearing in wells where $x<y$ increases the GaAs-like LO-mode frequency of $Ga_{1-x}In_xAs$, whereas tensile strain occurring in wells where $x>y$ decreases it, when compared to the respective strain-free frequencies. For a given layer, the frequency shift introduced by the biaxial strain produced by forcing both types of layers to have a common in-plane lattice paramenter is, following Cerdeira *et al.*,[15,22]

$$(\omega-\omega_0)/\omega_0=\delta\omega/\omega_0=-\beta\epsilon, \tag{5}$$

where ω and ω_0 are the Raman frequencies in the strained and strain-free material, respectively. The strain tensor in the layers is given by:

$$\epsilon_{xx}=\epsilon_{yy}=\epsilon, \quad \epsilon_{zz}=-\alpha\epsilon, \tag{6}$$

where $\epsilon=(a-a_0)/a_0$, and a and a_0 are the lattice constants of the strained layer and the unstrained material, respectively. The constant β is defined by

$$\beta=(\alpha p-2q)/2\omega_0^2 \tag{7}$$

with

$$\alpha=2C_{12}/C_{11}, \tag{8}$$

TABLE I. Parameters for bulk GaAs and InAs used in our calculation. Those for the $Ga_{1-x}In_xAs$ are originated by interpolation.

Material	P/ω_0^2	q/ω_0^2	α	β
GaAs	-1.7533^a	-2.4533^a	0.902^c	1.67^c
InAs	-0.9400^b	-2.0800^b	1.087^c	1.57^c

[a]From Ref. 37.
[b]From Ref. 15.
[c]From Ref. 38.

where p and q are phenomenological parameters describing the behavior of phonon frequencies in the presence of an homogeneous strain. According to these equations, the frequency shift of the LO-phonon mode is proportional to the layer strain. Since β is a positive magnitude, the negative (positive) frequency shift of the GaAs-like LO phonon indicates that tensile (compressive) strains are present in the $Ga_{1-x}In_xAs$ layers. The phonon coefficients (p,q) have been reported previously[15,37] and are listed in Table I together with α and β.

In Fig. 4 we present the calculated strain as a function of the difference in In content between wells and barriers, $x-y$. Since uniaxial stress measurements for the alloy material are not available, we have assumed that α and the phonon coefficients p/ω_0^2 and q/ω_0^2 vary linearly with composition between their respective values for the binary compounds. The precise concentration of In in each type of alloy layer was obtained from the x-ray measurements, as described before. As seen in Fig. 4 the strain changes linearly with the difference in In content. This result indicates that, in our samples, the lattice mismatch is accommodated by elastic strain up to a value as high as 3%. No clear difference between the 32 and the 40 Å well width samples was observed.

We have also estimated the critical thickness T_c of the layered system as a whole, above which the elastic energy accumulated in all the layers can be partially released by the formation of misfit dislocations. Following Hull *et al.*[6] this critical superlattice thickness is approximately equal to the

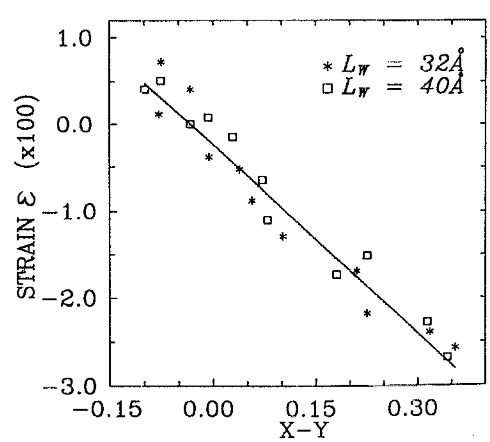

FIG. 4. Variation of the biaxial elastic strain vs In concentration difference $(x-y)$ between wells and barriers in the investigated $Al_{1-y}In_yAs/Ga_{1-x}In_xAs$ MQWs. The line is only a guide to the eyes.

critical thickness of a single alloy layer of an effective composition which in our case is given by

$$X_{\mathrm{eff}}=L_w(x-y)/(L_w+L_b), \tag{9}$$

where L_w and L_b are the well and barrier widths, respectively. This gives for the sample with the highest difference in In content (i.e., lower T_c) $T_c \simeq 2000$ Å which is clearly above the thickness of our layered structures (1120 Å). This supports our conclusion that the MQW system is commensurate with the substrate, and that the strain is only stored in the well layers, being zero in the barrier layers.

IV. CONCLUSIONS

Highly strained $Al_{1-y}In_yAs/Ga_{1-x}In_xAs$ ($y \simeq 0.1$) MQWs have been grown by ALMBE on [001]-GaAs substrates. Composition and layer thicknesses have been determined by means of DCXRD. Raman scattering has been used to monitor the stress present in these structures. We find that, when grown on a thick ($\simeq 1$ μm) AlInAs buffer layer with the same In content as the barrier material, the whole MQW system is commensurate with this buffer layer and the strain is only accumulated in the wells, for a wide range of In concentration ($0 \leqslant x \leqslant 0.44$). From the GaAs-like LO-phonon shift we obtain that in our samples the lattice mismatch is entirely accommodated by elastic tension up to a value of 3%. The epitaxial QW layers sustain then biaxial compression or tension accordingly, as their lattice constant is larger or smaller than that of the barriers. The versatility of these structures makes them very attractive for application in electronic and optoelectronic devices.

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