Relaxation behavior of undoped $\ln_x Ga_{1-x}P$ 0.5<*x*<0.7 grown on GaAs by atomic layer molecular-beam epitaxy

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We present a study of the relaxation behavior of compressive $\ln_x Ga_{1-x}P$ layers grown by atomic layer molecular-beam epitaxy at $T_s = 420$ °C with $x = 56\% \pm 3\%$ and $x = 67\% \pm 3\%$. Similar (thickness and composition) $\ln_x Ga_{1-x}P$ layers were grown under different growth conditions in order to assess the influence of the stoichiometry of the growth front on the structural properties and the relaxation process of this material system. All $\ln_x Ga_{1-x}P$ layers were characterized by double-crystal x-ray diffraction, transmission electron microscopy, and Nomarski interference. Our results show that surface stoichiometry during growth does not affect the relaxation behavior of $\ln_x Ga_{1-x}P$ layers but strongly determines their structural characteristics related to composition modulation features which appear in all our $\ln_x Ga_{1-x}P$ layers. We have established an empirical relation between residual strain and thickness. This relation makes predictable the residual strain of more complicated structures which can be introduced as buffer layers in lattice-mismatched heteroepitaxial systems. © 1996 American Institute of Physics. [S0021-8979(96)00218-6]

I. INTRODUCTION

 $In_xGa_{1-x}P$ alloy in the composition which matches the GaAs lattice parameter (x=0.48) has attracted in the past great attention for its application in visible light emitters and as an alternative to AlGaAs in GaAs-based devices.

This alloy has another interesting technological application as the constituent material in buffer structures to achieve any lattice parameter from that of GaP to that of InP, when convenient substrates as Si or GaAs are used. In fact, $In_rGa_{1-r}P$ alloys with very low mismatch (0.1%) have been recently used as intermediate layer between ZnSe epitaxial layers and GaAs substrates.¹ In order to fabricate good quality (flat and relaxed) buffer layers taking advantage of the wide lattice parameter range offered by this alloy, it is mandatory to know the plastic relaxation characteristics of this material in order to be able to predict its behavior in complicated designed structures. Up to now there has been no clear knowledge of the relaxation behavior of $In_rGa_{1-r}P$ layers. For example, different authors¹⁻³ have agreed that lowmismatch In_xGa_{1-x}P layers grown by different epitaxial techniques show a higher than expected residual strain. On the other hand, it is now well established⁴⁻⁸ that $In_{x}Ga_{1-x}P$ layers of composition close to the lattice matching condition of GaAs substrates (x=0.48) show quasiperiodic variations in composition over ranges from a few nm to hundreds of nm. This modulated phase separation due to spinodal decomposition results in lattice strains which produce contrasts in transmission electron microscopy (TEM) images. It is the aim of this work to study the influence on lattice relaxation of this inhomogeneous strain distribution. With that purpose we have studied the relaxation behavior of compressive $In_xGa_{1-x}P$ layers with x varying between $53\% \le x \le 59\%$ and $64\% \le x \le 70\%$ grown on GaAs (001) substrates by atomic layer molecular-beam epitaxy (ALMBE).⁹ This growth technique, together with *in situ* optical characterization by reflectance anisotropy measurements, is especially suitable for controlling the surface stoichiometry during growth. As composition fluctuations occur at the growth front, we have changed the stoichiometry during growth of the $In_xGa_{1-x}P$ layers trying to actuate on the phase separation process with the aim of studying the influence of these structural features on lattice relaxation.

Double-crystal x-ray diffraction (DCXRD) has been used to measure the alloy composition and the residual strain of the $In_xGa_{1-x}P$ alloys under study. All the layers were studied by TEM.

Our experimental results of strain versus thickness allow us to establish an empirical law which can be used to predict the strain state of the layers from their thicknesses in a similar way as in the case of other strained III–V semiconductors^{10–12} but with a higher than expected value of the critical thickness. TEM results clearly show a close correlation between growth conditions and composition modulation features which is not dependent on the strain in the layer. Some questions related to plastic relaxation of $In_xGa_{1-x}P$ alloy are briefly discussed at the end of this article.

II. EXPERIMENTAL PROCEDURE

All the $In_xGa_{1-x}P$ layers studied in this work have been grown at a substrate temperature of T_s =420 °C by ALMBE using a special phosphorus solid source with fast acting valve and cracking section.¹³ Semi-insulating GaAs (001) was always used as substrate and growth rate was 1 monolayer per second (ML/s).

 P_2 beam equivalent pressures (BEP) typically used in this work are about 2×10^{-6} Torr. Ga and In fluxes were previously calibrated by reflection high-energy electrondiffraction (RHEED) oscillations in GaAs and InP homoepitaxial growth runs by conventional MBE.

The growth process was in situ monitored by RHEED and reflectivity difference (RD) techniques. The reflectivity difference experimental setup has been described in detail elsewhere.¹⁴ In this work we have used a simplified experimental setup at the fixed wavelength of a He-Ne laser for monitoring in real time the surface stoichiometry changes during pulsed beam growth. At this wavelength, $\lambda = 6328$ Å, the reflectance difference for light polarized parallel to [110] and [110] directions supplies a signal (RD signal) of comparable amplitude for GaAs and other III-V compounds and their alloys¹⁴ and it is sensitive primarily¹⁵ to surface anisotropy induced by the group-III surface dimers along the [110] direction. The RD signal amplitude can be related to the density of group-III element dimers on the surface and therefore provides a measure of surface stoichiometry which changes periodically every monolayer growth cycle for ALMBE growth.¹⁶

In order to study the influence of surface stoichiometry on the growth mode and the relaxation process, different growth conditions have been used by changing the time duration of the P_2 pulses in the different growth runs.

Photoluminescence characterization was made on lattice-matched $In_xGa_{1-x}P$ layers to test the quality of this material when it is free of defects due to plastic relaxation.

All samples were characterized by DCXRD in order to obtain the alloy composition and the strain state in the $In_xGa_{1-x}P$ layers grown under different growth conditions.

Surface morphology of the $In_xGa_{1-x}P$ alloy layers has been observed by Nomarski optical interference microscopy. The layers were also examined by TEM in cross section and plan view. Samples were thinned by mechanical polishing and Ar⁺-ion milling. TEM observations were performed with a JEOL 1200-EX at accelerating voltage of 120 kV.

III. IN SITU EXPERIMENTAL RESULTS

Figure 1 shows the RD signal amplitude corresponding to $In_xGa_{1-x}P$ layers growth by ALMBE, together with the shutter sequence for the In, Ga, and P₂ effusion cells. Notice that Ga and In cells are always open during growth while the P₂ cell pulses once every second (growth rate is 1 ML/s).

The RD signal amplitude labeled "a" in Fig. 1 corresponds to the change of stoichiometry which occurs in every monolayer growth cycle under the given growth conditions. The RD signal amplitude labeled "b" in Fig. 1 corresponds to the full coverage of GaIn at the surface and is taken as a reference.

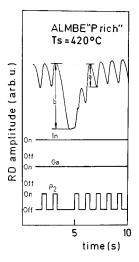


FIG. 1. Reflectivity difference (RD) signal amplitude observed during ALMBE growth of $In_xGa_{1-x}P$. The ratio r=a/b between (a) RD signal amplitude during growth and (b) maximum RD signal amplitude is defined as the stoichiometry parameter. The RD signal shown corresponds to P-rich conditions ($r \approx 0.4$). In the bottom part the shutter cells sequence is also shown.

By changing the duration of the phosphorus pulse during every monolayer growth cycle (typically from 0.1 to 0.3 s at 1 ML/s growth rate) we produce changes of the surface stoichiometry between different growth runs. In the following we use the ratio r=a/b as the stoichiometry parameter defining the different growth conditions used in this work; notice that *r* can change from 0 (no RD signal, growth under phosphorus saturation) to 1 (maximum change of stoichiometry in every layer without producing In and/or Ga droplets).

The RD signal record plotted in Fig. 1 corresponds to a value of the stoichiometry parameter r=0.4 which we consider as "P-rich" conditions. In this case surface reconstruction as observed by RHEED changed from 2×1 to a faint 2×4 in every monolayer. We have also explored other growth conditions corresponding to r=0.2, "P highly rich" conditions with a 2×1 surface reconstruction which remained constant during the whole monolayer growth cycle, and "GaIn-rich" conditions with a stoichiometry parameter in the range $0.7 \le r \le 0.8$. In this last case, surface reconstruction was 2×4 with variable intensity during the monolayer growth cycle. RHEED patterns showed that the growth conditions under study always preserved a monolayer-bymonolayer growth mode.

In Fig. 2 we show the PL spectrum at 10 K of a 1000nm-thick $In_xGa_{1-x}P$ layer lattice matched to GaAs grown by ALMBE with a stoichiometry parameter r=0.4. The peak energy and the width correspond to a high-quality disordered alloy of that composition,¹⁷ indicating that ALMBE at rather low substrate temperature ($T_s = 420$ °C) is a suitable growth technique for $In_xGa_{1-x}P$ alloys.

In order to study the $In_xGa_{1-x}P$ plastic relaxation behavior two series of samples with different In content, $x=56\% \pm 3\%$ and $x=67\% \pm 3\%$, have been grown. Each series consists of different thickness $In_xGa_{1-x}P$ layers with stoichiometry parameters in the range $0.2 \le r \le 0.8$. In this way we can obtain information about the influence of growth

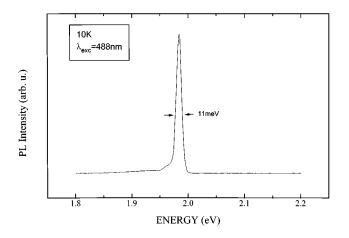


FIG. 2. Photoluminiscence spectrum taken at 10 K of a 1000-nm-thick $In_{0.47}Ga_{0.53}P$ layer grown by ALMBE on GaAs(001).

conditions on $In_xGa_{1-x}P$ relaxation behavior. Structural characterization results are shown in the following.

IV. STRUCTURAL CHARACTERIZATION (DCXRD, TEM, NOMARSKI)

A. DCXRD results

All samples were characterized by DCXRD in the (+ -)Bragg arrangement for the (004) reflection and in the $\theta+\Phi$, $\theta-\Phi$ arrangement for the (115) reflections. These four rocking curves were taken in the [110] and [110] directions in each sample. From the recorded data and by using a dynamical simulation program we have obtained the alloy composition and the strain state in the In_xGa_{1-x}P layers.

Tables I and II show the nominal values of layer thickness, the experimentally obtained In content, misfit strain ϵ_0 , in-plane strain obtained from DCXRD measurements both in [110] and [110] directions, and the stoichiometry parameter r of the In_xGa_{1-x}P layers studied in this work. The ratio between the nominal value of layer thickness d and its relax-

ation critical thickness $d_{\rm cr}$ is also shown. The $d_{\rm cr}$ value corresponds to the layer thickness at which strain relaxation starts in this material system, being determined by DCXRD measurements in this work.

Tables I and II correspond to samples with In content of $x=56\% \pm 3\%$ and $x=67\% \pm 3\%$, respectively. In the following we label the different samples starting with I if they appear in Table I or with II if they are in Table II. Layers are ordered by increasing thickness in both tables.

The mean value of the in-plane strain of $In_xGa_{1-x}P$ layers obtained from DCXRD measurements in [110] and [110] directions is plotted on Fig. 3 against thickness.

Data plotted in Fig. 3 show that relaxation behavior of $In_xGa_{1-x}P$ layers, $0.53 \le x \le 0.70$, is quite similar to that observed in $In_xGa_{1-x}As$ layers, $x \le 0.30$.^{10–12} As in this last case, strain remains constant with thickness until an appreciable (by x-ray measurements) strain relaxation takes place when the critical thickness d_{cr} is reached, $d_{cr}=K/\epsilon_0$ (ϵ_0 : misfit strain; *K*: constant). The strain at greater thickness than d_{cr} is $\epsilon(d) = K/d$. This expression describes strain versus thickness behavior for any alloy composition under study until a work-hardened regime is reached at large d ($d \ge 400$ nm) where the strained layers do not relax any further.

Error bars and experimental strain values for $In_xGa_{1-x}P$ layers below critical thickness with $x \approx 56\%$ have been omitted for clarity in Fig. 3 (see data in Tables I and II). The dashed line in Fig. 3 shows the $In_xGa_{1-x}As$ relaxation behavior previously studied in the literature.^{10,11} The corresponding horizontal dashed lines for $In_xGa_{1-x}As$ layers have also been omitted.

In the case of $In_xGa_{1-x}P$ layers we obtain experimentally that the value of the product ϵd is K = 1.4 nm (solid line in Fig. 3), while that reported for $In_xGa_{1-x}As$ layers was K=0.8 nm (dashed line on Fig. 3). This expression ($\epsilon d = 1.4$ nm) is valid for the values of residual strain measured in all the layers studied, indicating that growth conditions (r value) do not have a strong influence on the relaxation process of $In_xGa_{1-x}P$ layers.

TABLE I. Nominal thickness and experimental values of composition and strain obtained by DCXRD of $In_xGa_{1-x}P$ ($x=56\%\pm3\%$) layers grown by ALMBE under P-rich conditions (0.2 < r < 0.4) and GaIn-rich conditions (r=0.7). The last column shows the ratio between the nominal thickness and the experimental relaxation critical thickness. Values of misfit strain ϵ_0 calculated for the obtained alloy composition have also been included.

Sample	Nominal thickness (nm)	In Content DCXRD (%)	Misfit strain $\epsilon_0 (10^{-3})$	Strain DCXRD (10 ⁻³)	r	$d/d_{\rm cr}$
I-a	150	53	-3.4	$[\overline{110}]:-3.5\pm0.4$ $[110]:-3.4\pm0.2$	0.7	0.4
I-b	200	56	-5.5	$[\overline{110}]:-5.2\pm0.4$ $[110]:-4.3\pm0.3$	0.4	0.8
I-c	200	59	-7.8	$[\overline{110}]:-7.5\pm0.2$ $[110]:-7.0\pm0.5$	0.3	1.1
I-d	300	56	-5.5	$[\overline{110}]:-5.4\pm0.1$ $[110]:-5.7\pm0.3$	0.7	1.2
I-e	300	57	-6.3	$[\overline{110}]:-5.8\pm0.3$ $[110]:-4.3\pm0.3$	0.2	1.3
I-f	400	56	-5.5	$[110]:-4.9\pm0.2$ $[110]:-4.2\pm0.3$	0.2	1.5
I-g	500	57	-6.3	$[\overline{110}]:-5.1\pm0.4$ $[110]:-4.5\pm0.3$	0.2	2.3

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TABLE II. Nominal thickness and experimental values of composition and strain obtained by DCXRD of $In_xGa_{1-x}P$ ($x=67\%\pm3\%$) layers grown by ALMBE using P-rich conditions (0.4 < r < 0.5) and GaIn-rich conditions ($0.7 \le r \le 0.8$). The last column shows the ratio between the nominal thickness and the experimental relaxation critical thickness. Values of misfit stain ϵ_0 calculated for the obtained alloy composition have also been included.

Sample	Nominal thickness (nm)	In Content DCXRD (%)	Misfit strain $\epsilon_0 (10^{-3})$	Strain DCXRD (10 ⁻³)	r	$d/d_{\rm cr}$
II-a	42.6	67	-13.7	$[\overline{110}]:-13.2\pm0.5$ $[110]:-13.5\pm0.7$	0.4	0.4
II-b	71	68	-14.4	$[110]:-13.7\pm0.5$ $[110]:-13.8\pm0.4$	0.4	0.7
II-c	129	70	-15.9	$[110]:10.3\pm0.4$ $[110]:-9.7\pm0.4$	0.7	1.4
II-d	150	67	-13.7	$[110]: -8.8 \pm 0.5$ $[110]: -5.8 \pm 0.4$	0.8	1.4
II-e	200	66	-13.0	$[110]:-6.5\pm0.4$ $[110]:-5.3\pm0.5$	0.5	1.9
II-f	250	64	-11.5	$[110]:-4.6\pm0.4$ $[110]:-6.2\pm0.3$	0.5	2.1
II-g	400	65	-12.2	$[110]:-4.0\pm0.2$ $[110]:-4.4\pm0.4$	0.4	3.4
II-h	400	67	-13.7	$[\overline{110}]:-4.3\pm0.3$ $[110]:-5.6\pm0.4$	0.8	4.0

B. Surface morphology

All the layers grown under different growth conditions appear invariably mirrorlike to the naked eye. When viewed by Nomarski interference microscopy, a flat cross-hatched surface is always observed in samples grown under P-rich conditions while a faint cross hatch and some roughness appears in sample surface when GaIn-rich conditions were used. These results show that changes of stoichiometry during growth has some influence on surface morphology, although no differences in relaxation are appreciable by DCXRD measurements (see Fig. 3).

C. TEM results

A quasiperiodic microstructure with irregular periods varying from a few up to hundreds of nanometers is invariably observed in the $In_xGa_{1-x}P$ layers studied in this work, independent of composition, thickness, and, therefore, state of relaxation.

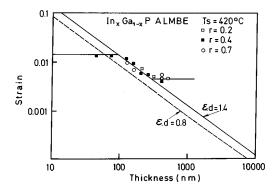


FIG. 3. Strain vs layer thickness for several $In_xGa_{1-x}P(0.53 < x < 0.7)$ layers grown by ALMBE under different growth conditions (0.2 < r < 0.8). The dashed line corresponds to the empirical relaxation law obtained for $In_xGa_{1-x}As$ (x < 0.3) (Refs. 10 and 11).

This microstructure appears, in agreement with previously reported data,^{6–8} under {220} reflection in cross section as a speckle contrast or as light/dark wavy lines running in the [001] growth direction with contrast modulation along the [110] directions in the (001) growth plane. Moreover, the alloy does not show any contrast modulation under {002} reflection in cross section. As has been suggested,^{6–8,18} these microstructural features are due to phase separation into regions of different compositions resulting in lattice strains which produce differences in elastic relaxation at the free surface of the samples prepared for TEM studies.

The kind of contrast observed, speckle type or dark/ bright lines, is present over the whole thickness of the layers with independence on misfit strain. In the case of latticemismatched layers these features appear when the layers are fully strained, remaining after relaxation by dislocation generation takes place. Our main result is that the type of contrast modulation can be precisely correlated with growth conditions, independent of other parameters such as composition or thickness.

As an example we show in Fig. 4 cross-section micrographs taken under {220} reflection of two $In_xGa_{1-x}P$ layers with the same composition and similar thickness but grown under P-rich conditions [Fig. 4(a)] and GaIn-rich conditions [Fig. 4(b)] (samples I-f and I-d on Table I, respectively). Fine speckle contrast is observed in the P-rich grown sample [Fig. 4(a)] while in the GaIn-rich grown sample [Fig. 4(b)] we observe a strong contrast modulation appearing as a columnar structure along the [001] growth direction, with a mean separation between fringes of about 80 nm. Both types of contrasts disappear under {004} reflection in cross section.

The columnar type of contrast is totally dependent on the growth front stoichiometry as it only appears in GaIn-rich grown samples. This demonstrates that modulation composition is originated at the growing surface and remains frozen

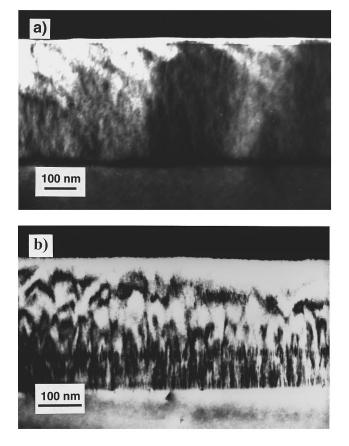


FIG. 4. Cross-section TEM micrographs taken at $\{220\}$ reflection of two In_{0.56}Ga_{0.44}P layers grown under (a) P-rich conditions and (b) GaIn-rich conditions [samples I-f and I-d in Table I correspond to (a) and (b), respectively).

inside the bulk as growth proceeds, as other authors have previously discussed.⁷

One important conclusion of our work is that the observed sharp contrasts due to composition variations, deleterious for the physical properties of the layers,⁶ can be suppressed by appropriately choosing the growth conditions. Other authors⁷ found some correlation between the wavelength of the composition modulation features with growth temperature. In our case, the layers grown by ALMBE, we can strongly actuate on the mobility of surface species at the growth front just by slightly changing the time of aperture of the phosphorus cell at any monolayer growth cycle. In this way we can influence, much more efficiently than changing growth temperature, the reduction or enhancement of phase separation at the surface.

Besides strain-induced contrast modulations, partially relaxed $In_xGa_{1-x}P$ alloy layers present 60° misfit dislocations at the interface. Figure 5 shows a plan-view image taken using the (220) reflection from sample I-f [see Table I for sample design and Fig. 4(a) for its cross section]. No threading dislocations and planar defects are observed in the epilayers while growing under P-rich conditions. When InGarich conditions are used during growth of InGaP layers planar defects are also observed and their density increases with the stoichiometry parameter *r*.

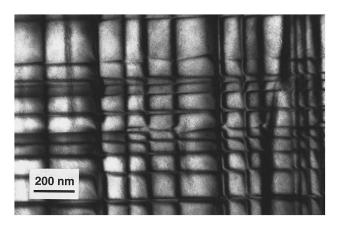


FIG. 5. Planar-view TEM micrograph from a 400-nm-thick $In_{0.56}Ga_{0.44}P$ layer. Observe the regular array of misfit dislocations. [See sample I-f in Table I for details and Fig. 4(a) for its cross section.]

V. DISCUSSION

The above exposed experimental results show that strain relaxation of InGaP layers depends on layer thickness according to the expression $\epsilon d = 1.4$ nm. Growth stoichiometry does not influence the relaxation behavior as long as a perfect 2D growth mode is preserved. The empirical law $\epsilon d = 1.4$ nm for relaxation of InGaP layers allows us to make predictions of the relaxation behavior of more complicated structures, providing a design tool for buffer layers.

There are, however, basic questions concerning InGaP relaxation which deserve some discussion, and for sure more experimental and theoretical work.

According to the model for relaxation of strained layers by Dunstan *et al.*^{10,11} plastic relaxation is predicted by the empirical relation $\epsilon d = 0.8$ nm, at least in III–V growth of the highest quality.¹² Based also on a large amount of experimental results from InGaAs alloys, these authors conclude that dislocation multiplication mechanisms are responsible for the observed empirical strain–thickness relationship.

Our experimental results show that an expression of the type $\epsilon d = K$ also describes the relaxation of InGaP layers pointing out similar strain relaxation mechanisms. However, we find that the constant value *K* is not universal but is material dependent, being larger in InGaP (K=1.4 nm) than in InGaAs (K=0.8 nm).

Two main differences between $In_xGa_{1-x}P$ layers, 0.53 < x < 0.70, studied in this work and $In_xGa_{1-x}As$ layers, x < 0.30,^{10–12} can be considered: first, the large difference in dislocation mobilities in the two binaries constituting these materials;¹⁹ second, the experimental observation of composition modulation in all the InGaP layers studied in this work.

A lower dislocation mobility in InGaP as compared to InGaAs should not be expected to account for these results, since no dependence of relaxation on temperature is found either in InGaAs (Ref. 10) or in InGaP (Refs. 1–3 and this work). However, we should expect that the composition fluctuations due to spinodal decomposition affect the dislocation multiplication mechanisms responsible for the observed relaxation behavior. In fact, it has been addressed for a long

time²⁰ that dislocations in a spinodally decomposed structure experience forces from the internal stresses and composition gradients.

We then could expect that in-plane strain/composition inhomogeneities have a considerable effect consistent with what we observe. It could be envisaged that small volumes of higher indium content and higher strain may not themselves relax (with respect to the surrounding material) because of their small size, while volumes of lower strain will act to inhibit multiplication mechanisms in which the dislocation is required to move through them. On this sort of model, the exact size of lateral inhomogeneities might not be too important, and what matters is that in all the samples observed here the contrast is found in one scale or another. We would still predict that laterally homogeneous InGaP alloy would relax with the same K as InGaAs, and what we observe here to raise the K to 1.4 nm is an athermal strengthening mechanism which should be also found in other alloys in the range of compositions which are not stable at the accessible epitaxial growth temperatures.

VI. SUMMARY AND CONCLUSIONS

We have studied the relaxation behavior of compressively strained $In_xGa_{1-x}P$ ($x=56\%\pm3\%$ and $x=67\%\pm3\%$) layers grown for the first time by ALMBE on GaAs(001) substrates under different growth conditions. Photoluminescence characteristics of InGaP layers lattice matched to GaAs indicate that ALMBE is a suitable technique for growth of high-quality phosphorus-containing III–V alloys.

We have found that growth stoichiometry does not strongly affect relaxation of InGaP layers, providing 2D growth mode is preserved. However, growth stoichiometry has a strong influence on the microstructure due to phase separation at the growth front.

We have obtained that abrupt composition variations can be suppressed by appropriately choosing the growth conditions. They also could be enhanced to make laterally confined quantum structures, as has been demonstrated in short period superlattice-based structures.²¹ Experimental results of strain ϵ versus thickness *d* have led us to establish an empirical law ($\epsilon d = K = 1.4$ nm) which allows us to predict relaxation of InGaP layers, and which can be used as a design tool for more complicated structures to be used in buffer layers. The constant value of the product ϵd obtained for InGaP layers (K=1.4 nm) is larger than that obtained for InGaAs layers (K=0.8 nm). The experimental evidence of different relaxation rates for different materials showing similar relaxation behavior (ϵd =K) points out that more work is needed for a better understanding of the behavior of strained alloys.

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