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STRAIN RELAXATION IN GRADED InGaAs AND InP BUFFER LAYERS ON GaAs (001)

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Abstract

We investigate compositionally graded $In_{x_0 \le x \le 0.5}Ga_{1-x}As$ and InP buffer layers which are prepared by molecular beam epitaxy on (001) GaAs substrate. The initial In content x_0 is equal to 0, 0.12, 0.18, 0.24, and 0.5 for the different samples. The In composition of the graded buffer increases linearly between x_0 and 0.5 with a fixed slope of 50% In-content per μm. The idea was to combine the advantage of surface flatness in homogeneous buffer layers and the reduced density of threading dislocations on the surface for graded buffer layers. The best compromise in terms of photoluminescence intensity and linewidth, electron mobility and crystal quality is achieved for $x_0 = 0.18$. For comparison to the InGaAs layers, we investigated also homogenous InP buffer layers on GaAs substrate. A strong photoluminescence peak with a linewidth of 5 meV is observed for 1 μm InP grown at 450°C applying a GaP decomposition source. The density of threading dislocations in the surface region is lower than in relaxed $In_{0.5}Ga_{0.5}As$ layers but still by far not as low as for the graded buffer layers.

Key Words: Graded buffer layers, strain relaxation, molecular beam epitaxy, GaInAs/GaAs, InP/GaAs, heterostructures, misfit dislocations, surface morphology, virtual substrate, transmission electron microscopy.

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Introduction

Strained semiconductor heterostructures are difficult to prepare because of thickness limitations due to strain relaxation. On the other hand, the strain can add considerable advantages to the device design, because it strongly influences the intrinsic properties like bandgap, band offset, carrier mobility, etc. Especially interesting is also the idea of using relaxed buffer layers as virtual substrate. Among the most commonly investigated strained material systems are ZnSe/GaAs, GaAs/Si, SiGe/Si, InGaAs/GaAs and, more recently, AlInGaAsP/GaAs.

Thick strain relaxed heterostructures usually suffer from high dislocation density in the surface region. A significant step towards lower defect densities has been achieved by applying thick compositionally graded buffer layers (Fitzgerald *et al.*, 1991; Inoue *et al.*, 1991; Krishnamoorthy *et al.*, 1992; LeGoues and Meyerson, 1991; Tuppen *et al.*, 1989). These linearly or step-graded buffer layers have shallow misfit gradients and are prepared at relatively high deposition temperatures which lead to instantaneous relaxation during the buffer layer growth. Accordingly, the resulting misfit dislocation network is distributed over the whole thickness of the graded part of the buffer. Pushed by the misfit stress, the threading dislocation segments can reach the substrate edges as pinning by intersecting dislocations is strongly reduced. This is in contrast to the situation in homogeneous buffer layers, where a huge number of misfit dislocations are usually nucleated after exceeding the critical thickness for pseudomorphic growth. However, for device applications, the graded buffer layers are often considered to be too complicated. The main problems are the total thickness and the corrugated surface morphology (Chang *et al.*, 1991) which often go along with a pronounced tilting of the relaxed buffer layer against the substrate (Ayers *et al.*, 1991).

In this contribution, we report on strain relaxed graded $In_{x_0 \le x \le 0.5}Ga_{1-x}As$ and InP buffer layers on GaAs. The goal is to combine the advantages of low defect density in graded buffer layers with acceptable

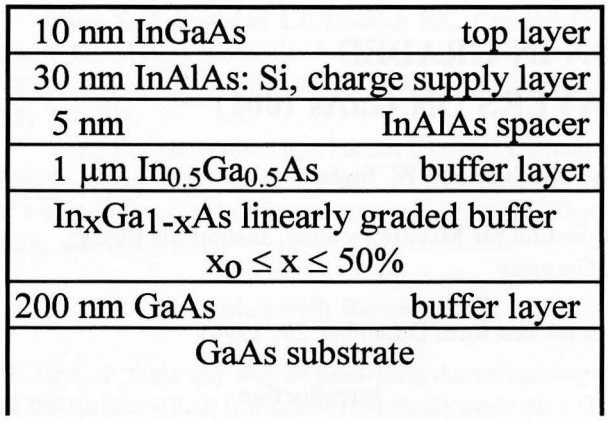

Figure 1. Schematic drawing of the layer composition. The In content of the graded $In_{x_o \le x \le 0.5}Ga_{1-x}As$ buffer layer starts at x_o at the GaAs interface and increases at a rate of 50% per micrometer.

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surface smoothness of strain relaxed layers with an abrupt misfit and a limited total thickness of less than 1 μm. The samples are characterized by double crystal X-ray diffraction (DCXRD), atomic force microscopy (AFM), photoluminescence (PL), and transmission electron microscopy (TEM). Modulation-doped InGaAs/AlInAs heterostructures are prepared on top of the buffer layers for Hall-effect measurements.

Experiments and Results

The samples are prepared by solid source molecular beam epitaxy (MBE). The In content of the InGaAs layers is determined *in situ* from reflection high energy electron diffraction (RHEED)-oscillations in pseudomorphic epilayers and *ex situ* with DCXRD on InGaAs/GaAs superlattice test layers. Additional In mole fractions are interpolated with respect to flux measurements of the In and Ga evaporation cells. The substrate temperature is measured with a W/Re thermocouple which was calibrated by using the oxide desorbtion temperature (580°C). The InP layers are prepared by applying a dimer phosphorus (P_2) molecular beam which is produced from a special GaP decomposition effusion cell. The source has a pyrolytic BN scavenger in front of the orifice of the crucible which traps the Ga flux. [For a detailed description see Shitara and Eberl (1994).] The semi-insulating GaAs substrates are nominally (001) oriented with a maximum miscut of about 0.1 degree.

Figure 1 shows a typical sample structure for the InGaAs buffer layers. After a 200 nm thick undoped GaAs film is grown at a substrate temperature (T_s) of 580°C, the undoped graded InGaAs layer is deposited with an In gradient of 50% per micron at T_s = 320°C. Within the graded layer, the growth process is repeatedly interrupted after every 200 monolayers to perform an annealing step at T_s = 420°C for about 60 seconds. The idea of this annealing is to move the threading dislocation segments and to smooth the surface. The surface smoothing is clearly observed with RHEED. The temperatures for growth and annealing are chosen in order to avoid In-segregation (Tournié *et al.*, 1992) or three-dimensional growth (Eckenstedt *et al.*, 1993) and finally to achieve optimum conditions for the preparation of smooth mirror-like buffer layers. The epitaxial growth is done under As-rich conditions with a (3x1)-reconstruction of the InGaAs surface and a ratio of group V to group III beam fluxes of about ten. A homogeneous 1 μm thick $In_{0.5}Ga_{0.5}As$ layer was deposited at 370°C on top of the graded buffer layer. A 5 nm thick InAlAs spacer layer was grown lattice matched to the InGaAs buffer, followed by a 30 nm Si-doped (about 1 x 10^{18} cm^{-3}) InAlAs charge supply layer and finally an undoped 10 nm InGaAs top layer.

Figure 2 shows cross-sectional TEM (XTEM) micrographs of: (a) the 1 μm thick $In_{0.5}Ga_{0.5}As$ buffer layer, (b) the graded InGaAs sample with x_o = 0.18, and (c) the sample with 1 μm thick InP layer. An extremely high density of threading dislocations (> 10^{11} cm^{-2}) is observed in the non-graded InGaAs sample in Figure 2a. In the sample with the graded InGaAs layer, we find the characteristic dislocation structure which is a small number of threading dislocations and many misfit dislocations running parallel to the surface separated across the whole area within the graded layer. No threading dislocations are observed in the region close to the surface. The strain relaxed InP buffer layer is shown in Figure 2c. The 1 μm thick InP layer was grown at T_s = 450°C on a 200 nm GaAs layer on (001) GaAs substrate. The sample grown at 450°C provides the best surface smoothness and PL linewidth as compared to similar InP buffer layers grown at higher or lower substrate temperatures. The growth rate was about 0.14 nm/sec and the P_2 pressure is about 4 x 10^{-6} Torr. The density of threading dislocations close to the surface is about two orders of magnitude lower than for the relaxed $In_{0.5}Ga_{0.5}As$ buffer layer, but far away from what is achieved for the graded layers where no threading dislocations are observed on the surface in TEM.

The surface structure is characterized by AFM. The left column in Figure 3 shows surface scans of 10 μm by 10 μm for the $In_{x_o \le x \le 0.5}Ga_{1-x}As$ buffer layers with x_o = 0.5, 0 and 0.18 and for the 1 μm InP buffer layer which is also described in Figure 2c. The right column shows the corresponding profile along the marked line. The vertical scale is extended by a factor of about 50. The surface of the relaxed $In_{0.5}Ga_{0.5}As$ layer shows a microscopic roughness, but is otherwise

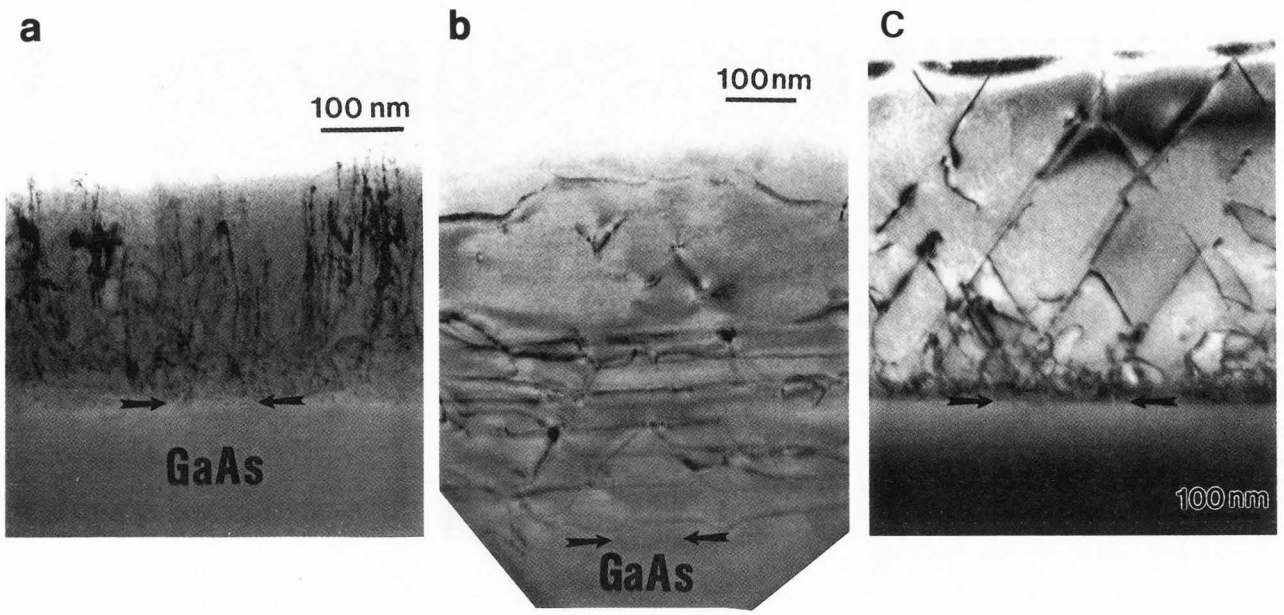

Figure 2. Cross-sectional TEM micrograph of (a) ungraded $In_{0.5}Ga_{0.5}As$ and (b) graded $In_{x_o \leq x \leq 0.5}Ga_{1-x}As$ buffer layer with $x_o = 0.18$ and (c) 1 μm InP on GaAs. The GaAs/InGaAs interface and the GaAs/InP interface are marked by arrows.

mirror-like without cross-hatching. The AFM-profile shows a corrugation with a maximum vertical amplitude of about 16 nm. The sample with the grading starting from zero In content has a pronounced cross-hatching observed with an optical microscope. The maximum vertical amplitude in the AFM-profile is about 110 nm. The best compromise of reduced surface corrugation with the strain relaxation mechanism typically observed for graded buffer layers is achieved in the sample with $x_o = 0.18$, having a maximum vertical amplitude of 23 nm. A totally different surface morphology is observed for the InP buffer layer. It shows a more islanded structure in AFM with a very low vertical amplitude comparable to the InGaAs sample with $x_o = 0.5$. The sample is optically mirror-like.

Figure 4 shows PL spectra for the $In_{x_o \leq x \leq 0.5}Ga_{1-x}As$ buffer layers with $x_o = 0$, 0.18 and 0.5. The PL measurements are performed at T = 8 K, applying a liquid nitrogen cooled Ge detector. For excitation, we use the 647 nm Kr^+ laser line. The band gap PL-signal originates mainly from the 1 μm thick $In_{0.5}Ga_{0.5}As$ layer on top of the graded buffer layer. The highest PL intensity and the smallest full width at half maximum (fwhm) is measured for the samples with $x_o = 0.12$ and 0.18. The spectrum of the sample with $x_o = 0.18$ with a fwhm of 18 meV is shown in Figure 4c. The ungraded sample (Figure 4a) shows no defined band pap luminescence. The sample with the grading starting from $x_o = 0$ is shown in Figure 4b and has a fwhm of 27.1 meV. For comparison, the typical linewidth measured in lattice matched $In_{0.53}Ga_{0.47}As$ layers on InP substrate is 3 meV (Sugawara *et al.*, 1991).

Figure 5 shows a PL spectrum for the InP buffer layer measured at 6 K. The linewidth of the InP peak at 1.42 eV is 5.3 meV. For comparison we have prepared an InP layer on InP substrate where we achieve a fwhm of 3 meV. The shoulder on the low energy side of the InP peak is probably related to the misfit defects within the strain relaxed layer. The peak at 1.49 eV originates from the GaAs substrate.

Table 1 summarizes the PL data together with DCXRD data and Hall effect measurements on modulation-doped InGaAs/AlInAs heterostructures. The DCXRD and Hall effect measurements are described in more detail in (Häusler *et al.*, 1995). The information extracted from the DCXRD measurements is the full width at half maximum of the peak originating from the strain relaxed $In_{0.5}Ga_{0.5}As$ and InP layer, and the tilting of the epilayer with respect to the GaAs substrate. The broadening of the peak is mainly due to the perturbation of the crystal by misfit defects. However, it does not directly reflect the defect density on the surface. The peak shape for the graded layers is also asymmetrically broadened due to the compositional grading itself. Consequently, it should not be directly compared to the homogeneous samples which are shown in the two right columns in Table 1. For the graded layers, the minimum fwhm is detected for the sample with $x_o = 0.18$.

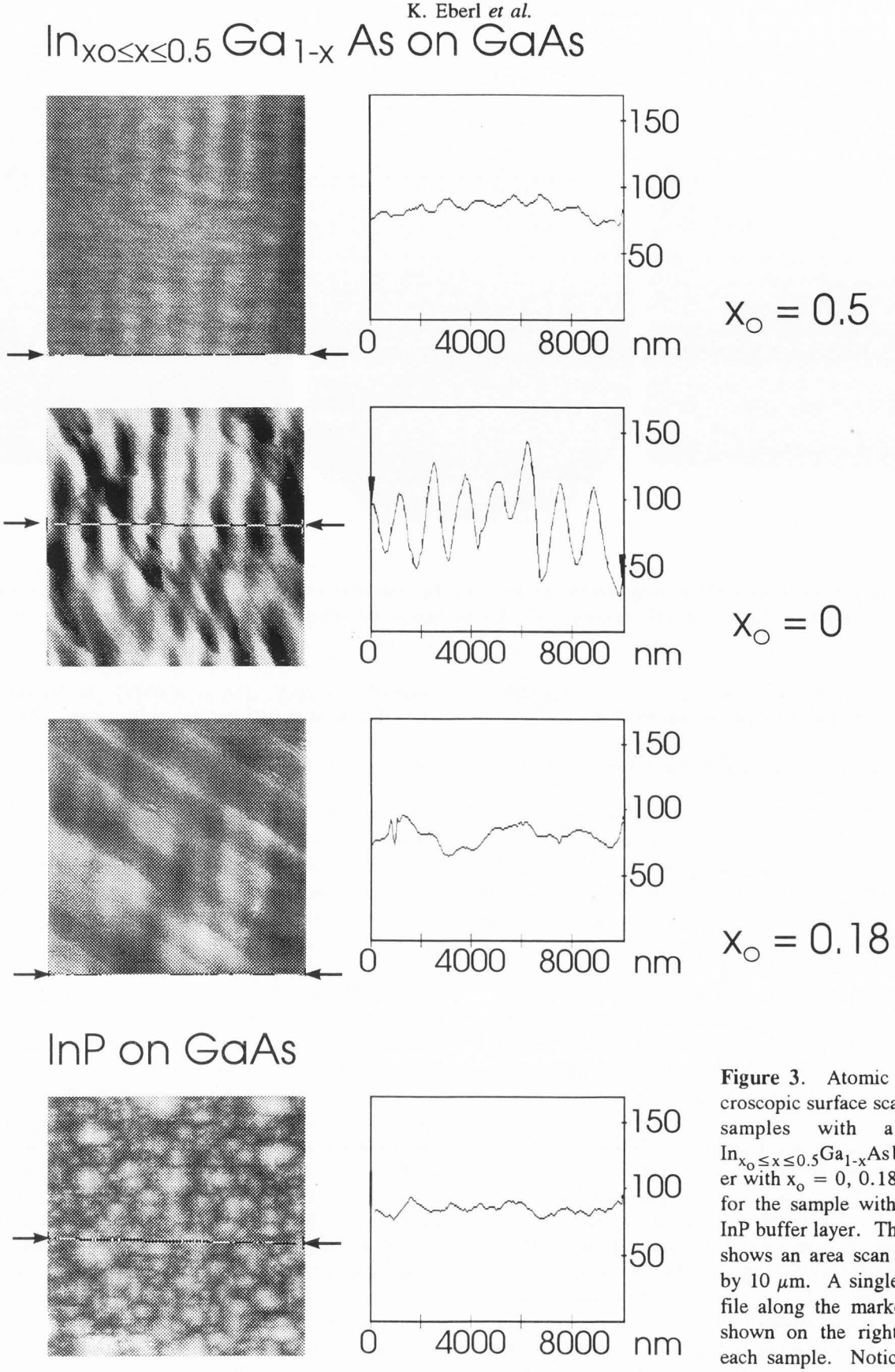

Figure 3. Atomic force microscopic surface scans for the samples with a graded $In_{x_o \le x \le 0.5}Ga_{1-x}As$ buffer layer with x_o = 0, 0.18, 0.5, and for the sample with an 1 μm InP buffer layer. The left part shows an area scan of 10 μm by 10 μm. A single line profile along the marked line is shown on the right side for each sample. Notice that the vertical scale is compressed by a factor of about 50.

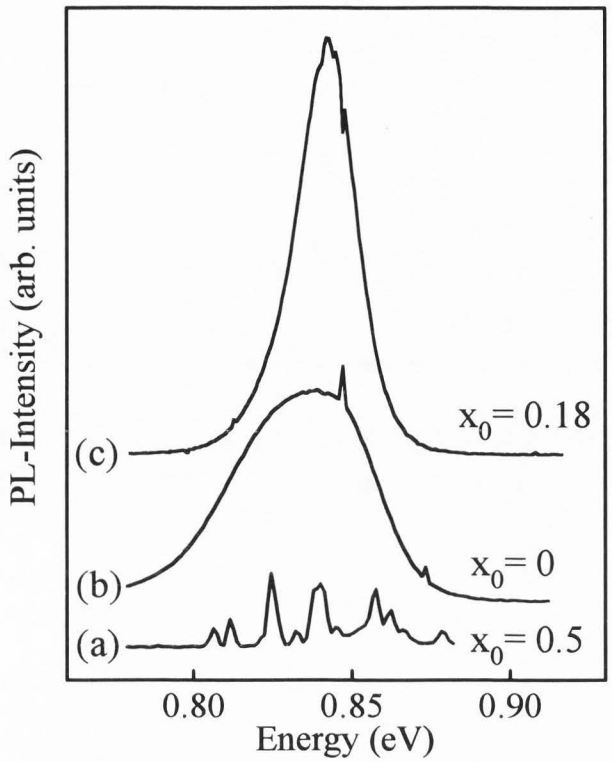

Figure 4. Photoluminescence spectra for the $In_{x_0 \le x \le 0.5}Ga_{1-x}As$ buffer layers with (a) $x_0 = 0.5$, (b) $x_0 = 0$ and (c) $x_0 = 0.50$. The PL signal originates mainly from the 1 μm thick $In_{0.5}Ga_{0.5}As$ layer on top of the graded layer (see Figure 1). The full width at half maximum for all samples are listed in Table 1.

The DCXRD signal form the InP sample is sharper as compared to the homogenous $Ga_{0.5}In_{0.5}As$ buffer layer which is probably due to the lower density of threading dislocations in the InP layer as shown in the transmission electron micrograph in Figure 2.

The misorientation of strain relaxed epilayers is well known (Ayers *et al.*, 1991; Ghandi and Ayers, 1988; Harmand *et al.*, 1989). We determined the tilt angle by evaluating the (004) X-ray rocking curves with the rotation angles of 0, 90°, 180° and 270° around the [001] axis normal to the (001) surface. The tilt angle increases with decreasing initial In fraction. The reason for the misorientation against the (001) substrate plane is a dislocation array with preferred direction of Burger's vectors (Eckenstedt *et al.*, 1993). No measurable misorientation is found for the homogeneous buffer layers $x_0 = 0.5$ and InP. Obviously, the investigated buffer layers with a larger initial misfit (larger x_0) cause a more symmetrically distributed nucleation of all possible misfit dislocations. LeGoues *et al.* (1992) have

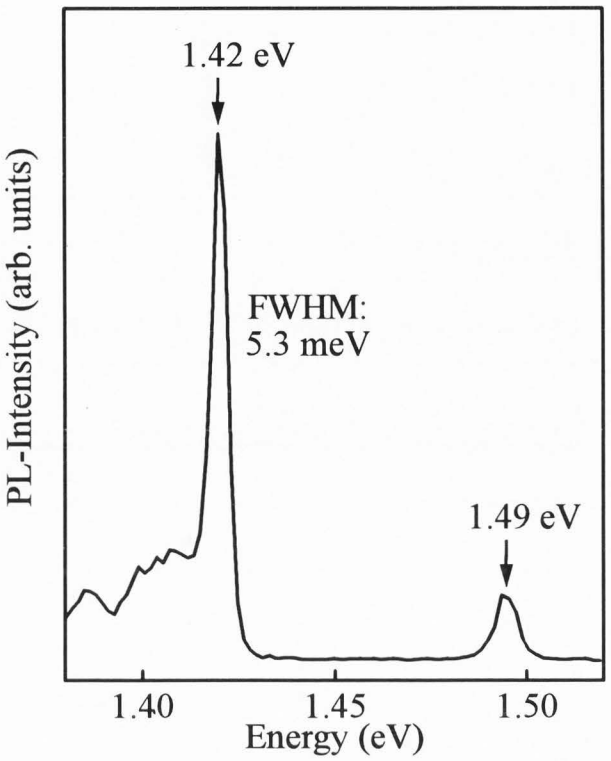

Figure 5. Photoluminescence spectra from the 1 μm thick InP strain relaxed buffer layer on GaAs (100) substrate.

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shown that internal multiplication processes can eject a large number of misfit dislocations with equal Burger's vectors out of one single defect. The accumulation of equal misfit dislocation can also causes a corrugation on the surface. This explains the large amplitude in the AFM linescan in Figure 3 for the sample with $x_0 = 0$, which has the largest tilt angle.

The idea for the samples with $x_0 = 0.12$ to 0.24 was to introduce a defined misfit step at the beginning of the graded layer which provides a more symmetrically distributed network of all possible misfit dislocations and than still apply the diluted dislocation distribution in graded buffer layers. The data presented in Table 1 and the TEM micrograph demonstrate that this concept can provide a buffer layer with low defect densities on the surface as well as fairly smooth surface morphology, especially for $x_0 = 0.18$.

The room temperature Hall effect data from modulation-doped InGaAs/AlInAs heterostructures are also included in Table 1. The measurements are performed using a Van de Paul geometry of 4 x 4 mm and by averaging the different current directions. For the sample with the InP buffer layer, we deposited an 0.5 μm thick

	$In_{x_0 \le x \le 0.5}Ga_{1-x}As$					InP
Initial In-content x_0	0	0.12	0.18	0.24	0.50	----
FWHM (mrad)	8.3	15.0	6.0	7.5	4.9	4.6
Tilt angle (mrad)	9.2	2.7	1.4	1.4	≈ 0	----
PL- linewidth (meV)	27.1	17.8	18.0	20.3	----	5.2
n_n ($10^{12}cm^{-2}$), 300K	1.34	1.82	2.85	1.79	1.69	1.69
μ_n (cm^2/Vs), 300K	7700	8700	9300	8980	3000	6700

Table 1. List of samples, including the corresponding DCXRD, PL, and Hall effect data.

$In_{0.5}Ga_{0.5}As$ layer followed by the same modulation-doped heterostructure as for the InGaAs buffer layers shown in Figure 1. A maximum two-dimensional electron density and mobility of 2.85 x 10^{12} cm^{-2} and 9300 cm^2/Vs, respectively, is achieved for the sample with x_0 = 0.18. The total thickness of the graded layer in this case is only about 0.64 μm. The electron mobility in the sample with the InP buffer layer is considerably higher than in the sample with the homogenous $In_{0.5}Ga_{0.5}As$ buffer layer.

Conclusions

In summary, we have shown that an initial step in the linearly graded InGaAs buffer layers on GaAs reduces the misorientation against the substrate, which also improves the surface smoothness considerably. The best PL linewidth and the electron density and mobility are achieved for x_0 = 0.18. The growth of the graded layers was performed at relatively low temperatures of T_s = 320°C with intermediate annealing steps at T_s = 420°C. The temperatures for growth and annealing are chosen in order to avoid In-segregation or three-dimensional growth and, finally, to achieve optimum conditions for reduction of the total thickness while still getting the diluted misfit dislocation structure which provides low dislocation densities on the surface.

The experimental results clearly demonstrate that a combination of an abrupt $GaAs/In_{x_0}Ga_{1-x_0}As$ heterostructure with x_0 about 0.2 and a linearly graded InGaAs layer combines the advantages of a reduced tilt angle in the epilayer, smooth surfaces, and low dislocation densities on the surface with a total thickness of the buffer layer below 1 μm.

The comparison of the homogeneous $In_{0.5}Ga_{0.5}As$ and the InP buffer layer shows that the relaxed InP layer provides superior optical and electronic properties. Further work will therefore be done in the application of the above concept to the GaInAsP compounds on GaAs.

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Discussion with Reviewers

K.L. Kavanagh: What are the specifications of the substrates?

Authors: The semi-insulating GaAs wafers are nominally (001) with the miscut smaller than 0.1 degree but with no defined miscut. We also checked the substrate miscut ourselves using a laser reflection technique during the DCXRD measurements for precise surface alignment. The experimental data are described in more detail in Häusler *et al.* (1995).

K.L. Kavanagh: Even though the buffers were grown and annealed at relatively low temperatures, are the authors sure that the growth was two-dimensional in all cases?

Authors: We are sure that we have two-dimensional growth in all cases because we did extensive RHEED studies during deposition in order to avoid island formation and three-dimensional growth mode.

K.L. Kavanagh: A pure edge dislocation will not generate epilayer tilt nor a surface step since it does not have a Burger's vector component perpendicular to the surface. Have the authors considered the possibility that the buffer layers with higher initial In concentrations have fewer and fewer 60° dislocations right at the start, hence causing the tilt to be reduced? This would also cause the surface roughness to be less since some or all of the roughness is created by steps generated by dislocations. Only 60° dislocations create a surface step.

Authors: We have not done a very detailed defect analysis with TEM. It is possible that the samples with $x_0 > 0$ have a larger number of pure edge type dislocations resulting in a reduced tilt and surface roughness. However, pure edge type dislocations do not have a glide system; thus, they usually appear when two appropriate 60° dislocations associate. That means that the statement which we made applies also in this case. Direct nucleation of pure edge dislocations at the interface is observed for extremely miscut substrates and in highly strained heterostructures like InAs/GaAs. In our case, the misfit step for the sample with $x_0 = 0.18$ is only 18% of what one has in the InAs/GaAs heterostructure.

K.L. Kavanagh: Do the authors have an explanation for why there is a "more systematic distribution of 60° Burger's vectors for the higher initial In concentration buffers" than for the lower? Do they agree with the simple model for an imbalance described in Ayers *et al.* (1991) (text reference) or do they have another explanation?

Authors: We did a very careful check on the question of whether the tilt was larger than the miscut by using an elaborated alignment technique to measure the substrate miscut and the epilayer tilt. The details are described in Häusler *et al.* (1995) (text reference). We are sure that the tilt is larger than the miscut for the sample with $x_0 = 0$. This can be explained by a dislocation multiplication process. An example was presented by LeGoues *et al.* (1992). There is also a very recent theoretical paper which explains this phenomenon for substrates which have a very small miscut [LeGoues *et al.* (1993) Appl. Phys. Lett. **62**, 140].

T.D. Lowes: What is magical about the starting point $x_0 = 0.18$? Is it because the layer is only 0.68 μm

thick or does it have to do with the degree of misfit at the interface, i.e., the magnitude of the so-called "abrupt misfit?"

Authors: As you already mentioned in your question, it is the degree of misfit at the interface. A considerably smaller misfit does not make so much of a difference compared to the graded layer starting form $x_0 = 0$, and a much larger misfit introduces too many threading dislocations; $x_0 = 0.18$ should also not be regarded as a very sharp optimum. The best data are in fact achieved for $x_0 = 0.15$ to 0.2, which is also stated in the text.

T.D. Lowes: Are the wafers miscut from the (001) or are they nominally (001)?

Authors: No, they are nominally (001) with the miscut smaller than 0.1 degree with no defined miscut. Intentionally miscut wafers are currently under investigation.

T.D. Lowes: How important is the annealing step after 200 nm of deposition? What is seen (XTEM for example) if the annealing step is not performed? How stringent are constraints on the temperature and time parameters for these annealing steps?

Authors: Similar samples grown without the annealing steps have a very rough surface structure. The time of the annealing intervals is not critical. Prolonged time does not further improve the layer quality.

J.-M. Baribeau: Have there been any attempts made (by asymmetrical reflection DCXRD, for example) to estimate the residual strain and what its consequence would be on the structural properties of the active layers?

Authors: We have done extensive X-ray diffraction measurements which include also the measurement of residual strain. The data have been published elsewhere (Häusler *et al.*, 1995). The residual strain is below 0.05%; thus, it does not significantly influence the properties of the active layer.

J.-M. Baribeau: Is it possible to establish any correlation between the magnitude of the surface ripples and the density of misfit dislocations?

Authors: This question is very significant and will fill many publications in the future. It can not be answered within the scope of the current paper. Our goal in this work was not to investigate the cause and detailed nature of the surface roughness. It was much more our aim to find out how we can reduce the surface roughness in order to achieve planar interfaces in the active layers.