Segregation in In$_x$Ga$_{1-x}$As/GaAs Stranski–Krastanow layers grown by metal–organic chemical vapour deposition

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Using quantitative high-resolution transmission electron microscopy we studied the chemical morphology of wetting layers in In$_x$Ga$_{1-x}$As/GaAs quantum dot structures which were optimized for applications to optical devices operating around 1.3 μm. The samples are grown by low-pressure metal–organic chemical vapour deposition on GaAs substrates. The In concentration profiles of the wetting layers are evaluated with the composition evaluation by lattice fringe analysis method. The profiles reveal a clear signature of segregation. A fit of the profiles with the Muraki et al. model for segregation reveals a segregation efficiency $R = 0.65 ± 0.05$ at the growth temperature of 550°C, which is significantly lower than segregation efficiencies observed in samples grown by molecular beam epitaxy at similar temperatures.

1. Introduction

Low-dimensional semiconductor materials have become one of the most active research fields in solid-state physics, chemistry and engineering. Quantum-well (QW) and quantum-dot (QD) structures produced by epitaxial growth techniques (mainly molecular beam epitaxy (MBE) and metal–organic chemical vapour deposition (MOCVD)) are of considerable technological interest, since they are used as active components in devices, for example in high-electron-mobility transistors and optoelectronics [1, 2]. In particular the In$_x$Ga$_{1-x}$As/GaAs material system is being studied intensively because of the possibility to form self-organized QDs by means of the Stranski–Krastanow growth mode under certain conditions. The development of QD lasers is expected to lead to increased quantum efficiency and to lower the threshold current density [3]. Properties of optoelectronic devices

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depend on the chemical morphology of the QWs or QDs forming the active region of a device. The growth of semiconductor nanostructures is governed by a complex and not fully understood interplay of surface and interface energies, formation of strain energy, elastic or plastic relaxation of strain energy, as well as by the kinetic growth processes such as adatom migration, interdiffusion and segregation.

Segregation is a known effect in MBE-grown In$_x$Ga$_{1-x}$As/GaAs heterostructures [4, 5] and leads to a strong enrichment of In on the growth surface compared with the underlying completed monolayers (MLs). Moison et al. [6] attributed the effect to the different chemical potentials of In and Ga atoms on the growth surface and suggested an exchange reaction of In and Ga in the topmost (‘surface’) layer and underlying (‘bulk’) layer. The change in the free energy per atomic reaction was called the ‘segregation energy’ $E_S$, and a value $E_S = 0.15$ eV was obtained [6] for In$_x$Ga$_{1-x}$As/GaAs at a growth temperature of 477°C. Gerard [7] published a method to obtain the amount $x_S$ of In on the growth surface by measuring a shift of the two-dimensional (2D)–three-dimensional (3D) transition of InAs when grown on a predeposited layer of In$_{0.08}$Ga$_{0.92}$As. He found that the amount of In on the surface of the predeposited layer with 8% In was well described by the segregation model suggested by Moison et al. [6] and obtained a segregation energy $E_S = 0.17$ eV for a growth temperature of 500°C. For In concentrations of the predeposited layer larger than 11% he observed a strong deviation of $x_S$ from the value predicted by the Moison et al. model. Toyoshima et al. [8] applied the method of Gerard to measure $x_S$ for In concentrations between 15 and 31%. These workers found that the amount $x_S$ of In in the ‘In floating layer’ is well described by the phenomenological segregation model suggested by Muraki et al. [9] also for nominal In concentrations larger than 11% where the Moison et al. model failed. Additionally, it was observed that the 2D–3D transition is correlated to the amount of In on the growth surface and occurs when $x_S$ reaches a value of more than 1.7 ML. Rosenauer et al. [10] measured In concentration profiles of wetting layers in buried InAs/GaAs Stranski–Krastanow structures by high-resolution transmission electron microscopy and found that the Muraki et al. [9] model is in agreement with the concentration profiles obtained. The amount of In in the floating layer, $x_S = 1.8$ ML (1.5 ML), was derived for growth temperatures of 530°C (480°C) using a fit of the Muraki et al. model to the experimental data. Evans et al. [11] applied temperature-programmed desorption measurements and found that the steady-state In floating layer contains 1.3 ML (1.6 ML) In for a growth temperature of 480°C (530°C) and that the surface-segregated In is liquid like in character. Garcia et al. [12] found by in-situ monitoring of stress acting on the GaAs substrate during the growth of In$_x$Ga$_{1-x}$As by MBE that the In floating layer is not chemically bonded to the crystal surface. Further indication to the mobile state of In atoms adsorbed in the In floating layer has recently been given by reflection high-energy electron diffraction (RHEED) experiments carried out by Martini et al. [13]. These workers showed that the exponential decrease in the RHEED oscillations observed at the onset of the In$_x$Ga$_{1-x}$As growth as well as the increase in the signal during cap layer growth is caused by scattering of the electron beam by mobile adatoms contained in the In floating layer. Larive et al. [14] and Kaspi et al. [15] observed
an increase in the segregation with increasing growth temperature, which is in contradiction to the Moison et al. [5] model. Dehaese et al. [16] suggested a kinetic model involving a two-energy-level system, which leads to the same segregation effect as the Moison et al. model for high growth temperatures above 500°C but additionally describes the kinetic limitation of segregation at a low temperature (400°C). As shown by Gerard [7], this model is therefore also limited to In concentrations below 11% for high growth temperatures. Additionally, the exchange reaction of In and Ga between the surface and subsurface layer does not account for the liquid character of the In atoms in the In floating layer evidenced recently [11–13], which limits $x_S$ to values smaller than or equal to 1 ML. Based on a recent energy-filtered transmission electron microscopy study of Walther et al. [17], Cullis et al. [18] developed a segregation-based model for the critical thickness of the 2D–3D transition. In agreement with Toyoshima et al. [8] Cullis et al. found that the 2D–3D transition occurs when the amount of In on the surface exceeds a critical value. Applying the segregation model of Dehaese et al. [16] to $\text{In}_{x}\text{Ga}_{1-x}\text{As}$ layers with nominal In concentrations of 25, 35 and 55% at a growth temperature of 540°C, a critical value of $x_S = 0.8$–0.85 ML In was obtained.

During growth of a GaAs cap layer, the In floating layer acts as a source for In atoms, leading to a significant incorporation of In atoms into the cap layer. Thus, segregation leads to a delayed incorporation of In atoms at the ‘lower’ interface between the GaAs buffer and $\text{In}_x\text{Ga}_{1-x}\text{As}$ layer, and to an exponential decrease in the In concentration within a GaAs cap layer at the ‘upper’ interface of the $\text{In}_x\text{Ga}_{1-x}\text{As}$ layer.

Whereas segregation is intensively investigated for MBE-grown structures, only little is known about segregation during MOCVD growth. Evidence for segregation taking place in atomic layer epitaxy using an MOCVD reactor was given by Arès et al. [19].

Here we present measurements of segregation efficiencies in MOCVD-grown $\text{In}_x\text{Ga}_{1-x}\text{As}/\text{GaAs}$ using transmission electron microscopy (TEM) techniques. We evaluate In concentration profiles of the wetting layer of buried QDs and fit the results with the segregation model given by Muraki et al. [9].

2. Experimental set-up

The samples were grown in a horizontal low-pressure MOCVD system (AIXTRON 200 AIX) at 20 mbar with trimethylgallium, trimethylindium solution and pure AsH$_3$ as source materials; palladium-purified H$_2$ with a flow rate of 7 standard l min$^{-1}$ was used as a carrier gas. The growth was performed on (001), exactly oriented semi-insulating GaAs substrates.

The investigated samples contain a Si-doped GaAs buffer layer 200 nm thick grown at a temperature of 750°C, a 1 μm Si-doped Al$_{0.37}$Ga$_{0.63}$As layer and a 80 nm GaAs layer, followed by an $\text{In}_{0.10}\text{Ga}_{0.90}\text{As}$ layer 5 nm thick, on top of which the deposition of the $\text{In}_x\text{Ga}_{1-x}\text{As}$ dots was carried out. The dots were covered with another 5 nm of $\text{In}_{0.10}\text{Ga}_{0.90}\text{As}$ and a GaAs cap layer 30 nm thick. Two different samples were grown. In sample A, the thickness of the $\text{In}_x\text{Ga}_{1-x}\text{As}$ QD layer was
4 ML and the nominal In concentration $x_0 = 0.55$, whereas sample B contains a 6 ML In$_x$Ga$_{1-x}$As QD layer with a nominal In concentration $x_0 = 0.50$. During the growth of the In$_x$Ga$_{1-x}$As layers, the growth temperature was fixed at 550°C and the growth rate at 1 ML s$^{-1}$. The AsH$_3$ partial pressures were kept at $1.4 \times 10^{-1}$ mbar and $2.8 \times 10^{-1}$ mbar for samples A and B respectively. The In-to-Ga flow ratio was kept constant. During the capping process the AsH$_3$ partial pressure was $5.7 \times 10^{-1}$ mbar.

TEM samples were prepared by standard methods. Plan-view specimens were obtained by grinding and polishing the substrate down to a thickness of about 20 μm, followed by Ar ion milling on the substrate side using an acceleration voltage of 4 kV and an incidence angle of 4°. Cross-section (001) and (110) TEM specimens were prepared by gluing two samples face to face, cutting slices, dimple grinding and Ar ion milling. A JEOL 4000EX microscope operating at 400 kV with a Scherzer resolution of 0.17 nm was used.

Composition profiles of the wetting layers are obtained using the composition evaluation by lattice fringe analysis (CELFA) method [20, 21]. (002) lattice fringe images are recorded by tilting the specimen approximately 3–5° around an axis perpendicular to both the electron beam direction and the interface plane. The excitation condition aimed at corresponds to a centre of the Laue circle (COLC) of (0, 20, 1.5) (a COLC of (0, 20, 2) would correspond to a strongly excited (004) reflection) and was adjusted by selected-area diffraction in a region including the investigated area or an area lying close it. The (002) beam is aligned parallel to the optical axis and the objective aperture is set around the (002) and (000) reflections. The CELFA method uses the following analysis steps. First, the image is subdivided into square-shaped image unit cells with a size corresponding to the 002 lattice plane spacing. For each image unit cell $n$, the (signed) modulus $A_{002}(n)$ of the 002 Fourier component of the image intensity is obtained by Fourier analysis. To account for variations in specimen thickness and tilts [22–24] within the image, we use the following approach: Bloch wave calculations of the modulus $A_{002}(x)$ of the 002 Fourier component of the image intensity is obtained by Fourier analysis. To account for variations in specimen thickness and tilts [22–24] within the image, we use the following approach: Bloch wave calculations of the modulus $A_{002}(x)$ of the 002 Fourier component of the image intensity is obtained by Fourier analysis. To account for variations in specimen thickness and tilts [22–24] within the image, we use the following approach: Bloch wave calculations of the modulus $A_{002}(x)$ of the 002 Fourier component of the image intensity is obtained by Fourier analysis. To account for variations in specimen thickness and tilts [22–24] within the image, we use the following approach: Bloch wave calculations of the modulus $A_{002}(x)$ of the 002 Fourier component of the image intensity is obtained by Fourier analysis.

By using parts of the image lying in the regions of the GaAs substrate or cap layer, a 2D third-order polynomial $A_{002}^0(n)$ is fitted to the values $A_{002}(n)$. The experimentally obtained normalized modulus is then compared with values calculated with the Bloch wave method. The Bloch wave calculations are based on knowledge of the Fourier components of the crystal potential. We compute the Fourier components of the crystal potential by density functional theory methods [25]. This is done to avoid the isolated atom approximation frequently used in TEM simulations. Additionally, the effect of static displacements [25, 26] occurring in ternary semiconductor material is also taken into account.
3. Experimental results

Figure 1 shows plan-view images from both sample A and sample B; the shape and size of the QDs are clearly comparable. The density of the QDs is 2.3 \times 10^{10} \text{cm}^{-2} for A and 7 \times 10^9 \text{cm}^{-2} for B.

Figure 2 reproduces colour-coded maps of the In distribution in the In$_x$Ga$_{1-x}$As WL evaluated with the CELFA method. Although care was taken during the exposure of the images to use regions where QDs were not visible, both images reveal small fluctuations in the In concentration within the In$_x$Ga$_{1-x}$As QW. Since these fluctuations occur not only in the central layer with high In concentration but also in the areas with nominally 10\% In, they are most probably caused by surface roughness or contamination caused by ion milling during the TEM specimen preparation, or by locally varying specimen tilt. Another possible origin could be the presence of small dots or parts of already-formed dots, depending on how the sample was cut during the TEM cross-sectional specimen preparation.

Figure 2 clearly evidences that the In concentration in sample A is a few per cent higher than in sample B, in agreement with the values of the nominal concentration ($x_{0,A} = 0.55$ and $x_{0,B} = 0.50$).

For quantification of the segregation efficiency, the concentration profiles along the [001] growth direction were obtained by averaging the evaluated In concentration along (002) lattice planes. The results are shown in figure 3 for both sample A and sample B (open circles). The figures reveal a clear signature of segregation.

The In concentration rapidly increases at the two lower interfaces, whereas it decreases exponentially at the two upper interfaces close to the cap layer. For evaluation of the segregation efficiency the measured profiles were fitted by the Muraki et al. model. To model different local nominal In concentrations ($x_0 = 0$ in the GaAs buffer and cap layers, $x_0 \approx 0.1$ in the In$_{0.10}$Ga$_{0.90}$As layer and $x_0 = 0.55$ (0.5) in the...
Figure 2. Colour-coded maps of the In concentration in samples A and B, evaluated with the CELFA method.
Figure 3. In concentration profiles obtained from samples A and B. The growth direction is from left to right. The error bars give the standard deviation encountered by averaging local concentration values along (002) lattice planes. The solid curves show the values of the concentration profile $x_b(n)$ as obtained from a fit of the experimental data with equation (1). The dotted curves correspond to the In concentration $x_0(n)$ (see equation (1)).
QD layer in sample A (B)), we used the following iterative formulation of the Muraki et al. model:

\[
\begin{align*}
(i) & \quad x_b(n) = x_s(n) + x_0(n), \\
(ii) & \quad x_s(n + 1) = x_b(n)R, \\
(iii) & \quad x_b(n) = x_b(n)(1 - R), \\
\end{align*}
\]

where \(n\) is the number of MLs, \(R\) is the segregation efficiency, \(x_b(n)\) is the In concentration in the upper crystal ML after growth of the \(n\)th ML, and \(x_s(n)\) is the In concentration in the In floating layer after growth of the \(n\)th ML. In the literature (see, for example, [7]), the amount of In in the floating layer is given in units of MLs of In, where 1 ML of In corresponds to \(x_s(n) = 1\). In the following, we shall use both notations. Equation (1) consists of the following steps.

(i) After growth of the \((n - 1)\)th ML, all In atoms of the In floating layer are incorporated into the upper crystal layer (corresponding to \(x_b(n)\)) and also the In atoms from the In source are added (corresponding to \(x_0(n)\)).

(ii) Then, a fraction \(R\) of the In atoms in the upper crystal layer segregate to the new floating layer (corresponding to \(x_s(n + 1)\)).

(iii) The amount of In in the upper ML is decreased by the fraction of In atoms that segregated into the floating layer.

In equation (1), the segregation efficiency \(R\) as well as the position and thickness of the layers were used as fitting parameters. The result of the fit is also shown in figure 3. The solid curve corresponds to the fitted concentration profile \(x_b(n)\), and the dotted curve indicates \(x_0(n)\). Obviously, the measured concentration profiles can well be fitted by the Muraki et al. model, clearly revealing that segregation is also present in samples grown by the MOCVD process.

From the fit of the Muraki et al. model to our concentration profiles we found that the segregation efficiency is \(R = 0.65 \pm 0.05\) for both sample A and sample B. For the thickness of the central layer we obtained 3 ML for sample A and 5 ML for sample B, compared with the nominal values of 4 ML and 6 ML respectively.

4. Discussion

Although the fitted concentration profiles are generally in fair agreement with the experimental profiles, significant deviations are observed in both samples at the top of the central In\(_x\)Ga\(_{1-x}\)As layer where the In concentration is high. In sample A (B), the measured In concentration is approximately \(\Delta x = 7\% (10\%)\) below the value of the fitted profile. This deviation can be explained by different effects.

Firstly, it can be an artefact of the measurement of the In concentration by the CELFA method. If the specimen thickness assumed for the CELFA evaluation (30 nm) is wrong, the evaluation contains errors that increase with increasing In concentration. As the CELFA technique uses a simple imaging condition where the relevant information (the local composition) is almost solely transferred by only one reflection (the chemically sensitive 002 reflection) centred on the optical axis,
objective lens defocus related artefacts are negligible in a wide defocus range between approximately $-150$ and $50$ nm [27].

Secondly, the $\text{In}_x\text{Ga}_{1-x}\text{As}$ layers are strained. In a thin TEM specimen, compressively strained layers are able to expand close to the sample surface, leading to bending of lattice planes [28, 29]. This effect strongly influences the intensity of the diffracted beams, because it leads to a local variation in the excitation errors.

To estimate the influence of the specimen thickness uncertainty as well as the effect of strain, CELFA evaluations of simulated images were carried out. Although the specimen thickness in the experiments was estimated to be smaller than 70 nm from a comparison of the island density in the image with the density obtained by plan-view TEM, images were simulated for a specimen thickness between 10 and 200 nm. The COLC aimed at in the experiment was (0, 20, 1.5). To account for the effect of local tilt variation, which also has been reported in the literature [23, 24], excitation conditions were varied corresponding to a COLC between (0, 20, 0) and (0, 20, 3). The local strain field was obtained by finite-element (FE) calculations. The FE models were generated according to the concentration profile (solid curve in figure 3 for sample B). Using the column approximation, the Howie–Whelan equations [30] were solved with the fourth-order Runge–Kutta method and the complex amplitudes of diffracted beams were calculated. The product of the amplitudes of the undiffracted and the 002 beam corresponds to the amplitude $A_{002}$ of the 002 Fourier coefficient of the image intensity for our imaging conditions. Concentration profiles along the growth direction were evaluated from the profiles of $A_{002}^N$ according to the CELFA method. In analogy to the evaluation of the experimental images, the simulated images were evaluated assuming a specimen thickness of 30 nm and a COLC of (0, 20, 1.5). As an example, figure 4 (left-hand axis) shows $A_{002}^N$ simulated for 70 nm specimen thickness and a COLC of (0, 20, 1.25). The variations in $A_{002}$ in the regions below 0 nm and above 15 nm are caused by lattice plane bending, which mainly affects the modulus of the undiffracted beam. The dashed curve is the fitted polynomial $A_{002}^0$ used to obtain normalized values $A_{002}^N$. Figure 4 (right-hand axis) also shows the In concentration evaluated from $A_{002}^N$, where a specimen thickness of 30 nm and a COLC of (0, 20, 1.5) were assumed. For comparison, the solid curve represents the true In concentration profile used as input for the simulation. Objective lens aberrations were not taken into account, because we found that their influence can be neglected at the imaging conditions applied [27]. To demonstrate that, for the In concentration profile used here, figure 5 shows the original profile of $A_{002}$ (same as in figure 4), and profiles where the effect of lens aberrations were taken into account. Because of damping by the transfer function of the objective lens, the original profile appears enlarged. All the profiles computed for defocus values between $-500$ and $+100$ nm are very similar. Only the parts below an abscissa of 0 nm and above 15 nm are affected slightly. This effect is due to defocus-dependent delocalization of the undiffracted beam with respect to the 002 beam. Figure 6 gives an overview of the results of our simulations. The maximum In concentration detected at the top of the central $\text{In}_x\text{Ga}_{1-x}\text{As}$ layer is plotted versus the specimen thickness for the different specimen orientations. One can clearly see that the effect of specimen thickness uncertainty and strain fields yield measured maximum In concentrations that are slightly too high in most cases. Only for few certain combinations of specimen thickness and
specimen orientation is a decreased value of the measured maximum concentration encountered. The deviations are smallest for a COLC of (0, 20, 1.5) that we aimed at in the experiment. The lower straight solid line in figure 6 corresponds to the maximum In concentration measured in the case of sample B, clearly lying below the values obtained by the simulation. Therefore, it seems unlikely that these effects are responsible for the deviation between the fitted segregation profile and the measured concentration profiles.

A third reason for the deviations, which seems more likely, is based on the presence of islands. Here we assume that the amount of In atoms contained in the islands is missing in the wetting layer. This could explain why the thickness of the wetting layers are approximately 1 ML smaller than the nominal values. We assume that the islands are formed owing to migration of In atoms from the wetting layer into the island. In addition, In atoms could also be provided from the In floating layer. This, however, seems unlikely, because a depletion of the In floating layer would result in an abrupt upper interface of the central In$_{x}$Ga$_{1-x}$As layers.

In contrast, the central In$_{x}$Ga$_{1-x}$As layers can be fitted with the same segregation efficiency as the In$_{0.1}$Ga$_{0.9}$As layers.

In layers grown by MBE, the 2D–3D transition occurs as soon as the amount of In in the floating layer exceeds approximately 1.6 ML [8]. Figure 7 shows the amount $x_s(n)$ of In atoms in the In floating layer as obtained from equation (1), plotted versus the number of MLs grown. Figure 7 shows that the amount of In
Figure 5. Profiles of the signed modulus $A_{002}$ for a specimen thickness of 70 nm and a COLC of (0, 20, 1.25). The grey curve was computed without lens aberration. Aberrations were taken into account for the other curves, which were calculated for different values of the defocus. The imaging parameters used are as follows: spherical aberration constant, 1.0 mm; beam semiconvergence, 1 mrad; defocus spread, 10 nm.

Figure 6. Maximum In concentration on the top of the central $\text{In}_{x}\text{Ga}_{1-x}\text{As}/\text{GaAs}$ layer obtained by the CELFA evaluation of images simulated for different specimen thickness and specimen orientation (the COLC is (0, 20, $L$), where $L$ is given in the key). For the CELFA evaluation, a specimen thickness of 30 nm and a specimen orientation corresponding to a COLC of (0, 20, 1.5) were assumed. The upper straight solid line shows the true In concentration assumed for the simulation. The lower straight solid line shows the maximum In concentration that we obtained in the experiment.
in the floating layer was less than 1 ML at the onset of the 2D–3D transition for both sample A and sample B. This finding is in clear contrast with the behaviour of samples grown by MBE.

Our MOCVD samples reveal a segregation efficiency \( R = 0.65 \pm 0.05 \) at a growth temperature of 550°C. Figure 8 compares this value with segregation efficiencies measured in MBE-grown samples. It clearly reveals that the segregation efficiencies of our MOCVD samples is significantly smaller than those observed in MBE-grown samples.

Figure 7. Profiles of the amount \( x_s(n) \) (see equation (1)) of In in the floating layer during growth, as obtained from the fit of equation (1) to the concentration profiles measured. The upper graph corresponds to sample A and the lower graph to sample B. (Note that, in the literature (see, for example, [8]), \( x_s(n) \) is expressed in units of MLs of In. In their notation, 1 ML of In corresponds to \( x_s(n) = 1 \).)
samples at similar temperatures. The difference between MBE- and MOCVD-grown samples can be explained by the different conditions and epitaxial processes at the growth surface. It can be speculated that the flux of the H\textsubscript{2} carrier gas along the specimen surface interferes with the formation of an In floating layer which is only weakly bound to the growth surface. A similar influence could be exerted by the large metal–organic molecules that are used to transport the Ga and In atoms on to the growth surface.

5. Conclusion

We measured In concentration profiles of wetting layers in MOCVD-grown In\textsubscript{x}Ga\textsubscript{1-x}As heterostructures buried in GaAs. The profiles clearly reveal that segregation of In takes place during growth. The fit of the concentration profiles with the Muraki et al. model of segregation yield a segregation efficiency $R = 0.65 \pm 0.05$ at a growth temperature of 550°C. This value is considerably smaller than the efficiencies evaluated in MBE-grown samples at a similar temperature. The difference between MBE- and MOCVD-grown samples is tentatively explained by the flux of the carrier gas and the presence of large metal–organic molecules at the growth surface in the case of MOCVD-grown samples, which could hinder the formation of an In floating layer that is only weakly bound to the growth surface. The In concentrations measured on top of the wetting layers are lower than expected from the Muraki et al. model, and the thickness of the wetting layers is approximately 1 ML less than expected from the growth conditions. This deficiency of In in the wetting layers is explained by a surface migration of In atoms from the wetting layer into the In\textsubscript{x}Ga\textsubscript{1-x}As QDs during growth.
Applying the segregation model of Muraki et al., we estimate that the amount of In in the In floating layer is approximately 0.85 ML at the onset of the 2D–3D transition. This value is significantly smaller compared with that for MBE-grown samples, where the growth mode transition occurs when the amount of In in the floating layer reaches 1.6 ML.

References