On the incorporation of indium in InAs-based quantum structures

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Abstract. Transmission electron microscopy (TEM) was applied to study In segregation during molecular beam epitaxy (MBE) growth of InAs-based quantum-dot (QD) structures with a focus on achieving QD luminescence at the technologically relevant wavelength of 1.3 μm. Quantitative composition analyses were carried out with the composition evaluation by lattice fringe analysis (CELFA) technique and In-segregation efficiencies were determined on the basis of atomic-scale composition profiles. The effect of the InAs-deposition rate on the In distribution was studied in detail which needs to be low, in the 0.005 ML/s regime to achieve long-wavelength emission. Further minimization of In-segregation induced intermixing can be achieved by lowering the substrate temperature during GaAs cap layer growth. Finally, a procedure is presented to extract the real composition of QDs taking into account their three-dimensional morphology and embedding in a GaAs cap layer.

1. Introduction

The properties of InAs/GaAs quantum-dot (QD) structures continue to be of interest due to numerous applications that have emerged during the past decade. In addition to established (opto)electronic devices like laser diodes and single-photon emitters [1,2] new applications in semiconductor spintronics are envisaged where InAs QDs could be promising for future devices in quantum information processing [3,4]. Selforganized QD formation is based on the Stranski-Krastanow (SK) growth mode which is characterized by a transition between two-dimensional (2D) layer and three-dimensional (3D) island growth. The critical InGaAs thickness for the 2D/3D growth-mode transition has been studied excessively over about two decades. While the SK growth was first solely attributed to strain relaxation in lattice-mismatched heterostructures, more recently the amount of segregated indium on the growth surface was discussed as a decisive factor for the growth-mode transition [5-7].

The resulting structures always consist of a continuous wetting layer (WL) and islands. However, the chemical composition of the WL and islands and QD properties such as size, shape and density are strongly dependent on the epitaxy conditions and deposited amount of InAs as demonstrated in comprehensive photoluminescence (PL) and transmission electron microscopy (TEM) studies, e.g. [8,9]. Segregation was recognized as an important mechanism in epitaxial growth of semiconductor...
nanostructures which may lead to a significant difference between the real and intended composition as already demonstrated in the pioneering work of Moisson et al. [10] for AlGaAs and Muraki et al. [11] for InGaAs. Muraki et al. proposed a phenomenological model which was shown to be well suited for the description of experimentally derived composition profiles of InGaAs quantum wells and wetting layers in InAs QD structures [7, 12]. In this model, the segregation efficiency $R$ characterizes the fraction of In atoms which segregate from the uppermost crystalline InGaAs layer into the layer deposited on top which is considered as a weakly bonded floating layer. The segregation efficiency depends on the temperature but also on other parameters like - in the case of molecular beam epitaxy (MBE) growth - the V/III-flux ratio, surface reconstruction and the type of As-source molecule (As$_4$ or As$_2$). High $R$ values between 0.65 and 0.93 are observed in the temperature interval between 370 and 560 °C according to [13] and references therein.

Strong In segregation in InGaAs has consequences with respect to achieving luminescence of QD-based laser diodes in the technologically interesting 1.3 and 1.5 µm range which requires large QDs (small confinement energies) with the highest possible In concentration. Optimum MBE conditions for PL with a peak at 1.3 and 1.5 µm are obtained at very low growth rates around 0.005 monolayer (ML)/s, temperatures around 500 °C and a deposition of 2 to 4 ML InAs which favours the formation of large islands [14, 9]. However, the high substrate temperature $T_s$ is not well suited to minimize segregation. Since lowering of $T_s$ leads to a reduction of the QD size [9], different strategies are used to optimize the QD structures with respect to long-wavelength emission as e.g. capping by InGaAs instead of GaAs [15, 16] or embedding of InAs QDs in InGaAs quantum wells [17].

Although TEM is frequently applied for the characterization of InAs-based quantum structures, quantification of the composition is scarcely found in literature even though of the group-III element distribution is essential for the understanding of the growth mechanisms and device properties. Techniques like electron energy loss spectroscopy [5, 6, 18] and quantitative scanning tunnelling microscopy [19] have been used to analyze the composition of InGaAs QD structures but quantification on the basis of the chemically sensitive {200} reflections is particularly suited in the case of InGaAs. Quantitative composition analyses on an atomic-scale were carried out in this study by applying the composition evaluation by lattice fringe analysis (CELFA) technique [20] which works well for planar InGaAs quantum-well type structures. However, quantitative analyses of QDs have been hampered up to now by the 3D morphology of the QDs which are embedded in a GaAs matrix. In a TEM cross-section sample with finite thickness, the measured In concentration will be inevitably too low for QDs due to an averaging effect along the electron beam.

In this study we compare in §3.1 InAs QD samples which were grown under identical MBE conditions apart from the InAs deposition rate. The reduction of the InAs deposition rate to a low value of 0.0056 ML/s is essential for achieving a PL peak at 1.3 µm. In §3.2 we will present a procedure that allows the correction of averaged composition data of InGaAs QDs embedded in GaAs which is accomplished by the determination of the sample thickness and QD shape. Finally, results on the effect of lowering the substrate temperature $T_{cap}$ during GaAs cap-layer growth are presented in §3.3. The latter experiments are motivated by the aim to reduce undesired In-segregation induced intermixing during overgrowth to eventually shift the PL towards even longer wavelengths.

2. Experimental techniques

All investigated samples were grown in a Riber Compact 21 MBE system equipped with effusion cells for Ga and In, and a valved cracker cell for As on GaAs(001) substrates covered by a GaAs buffer. The GaAs buffer growth was interrupted to reduce the substrate temperature from 580 °C to the value chosen for InAs deposition. Several sample series were investigated as outlined in detail in [9]. With respect to achieving 1.3 µm luminescence we focus here on the effect of the InAs deposition rate which was varied between 0.0056 ML/s (sample denoted as $S_{low}$) and 0.08 ML/s (sample $S_{high}$) with an As:In beam-equivalent pressure ratio (BEPR) of 80:1, As$_4$ as an As precursor and an optimized value for $T_{cap}$=500 °C. The amount of deposited InAs was 2.4 ML and a growth interruption of 10 s was introduced before the GaAs cap-layer growth.
In §3.3 we present results of a sample series where the substrate temperature was lowered after the InAs deposition (2.4 ML InAs, deposition rate 0.09 ML/s, As:In BEPR = 10:1, $T_s$=485 °C). Three samples were fabricated with identical InAs QD layers but different temperatures $T_{\text{cap}}$ during the deposition of the 50 nm GaAs cap layer. The capping temperature was 485 °C for the first sample. The initial 20 ML of the GaAs cap layer of the second sample were deposited at 410 °C before the temperature was raised to 485 °C. In the third sample, $T_{\text{cap}}$ was lowered to 350 °C for the first 20 ML and the rest of the GaAs cap-layer growth took place at 570 °C.

TEM cross-section samples were prepared along the [010]-zone axis using the procedure outlined by Bravman et al. [21]. Plan-view samples were obtained by chemical etching from the substrate side using a solution of NaOH (1mol/l) and H$_2$O$_2$ (30%) with a ratio of 5:1 to prevent defect formation during sample preparation.

TEM micrographs were recorded using a 200 keV Philips CM 200 FEG/ST transmission electron microscope. The determination of the In concentration with CELFA is based on HRTEM lattice-fringe images taken under {200} two-beam conditions close to the [010]-zone axis. Choosing an <100>-type zone-axis instead of a <110>-zone axis is essential to avoid contributions of the {111} reflections due to dynamical electron diffraction and nonlinear image formation. The selected [200] reflection is centered on the optical axis of the electron microscope to minimize delocalization. The local In concentration is determined by evaluating the local amplitude of the {200} Fourier component of the image intensity which is normalized with respect to the respective Fourier component in the adjacent GaAs. The local normalized {200} amplitudes are then compared with values calculated by the Bloch-wave method using structure factors according to [22] which comprise the effect of static atomic displacements in InGaAs. Due to the strong bending of the (002) planes in the QD area we use the (200) planes for the lattice-fringe imaging in §3.2. These planes are oriented perpendicular to the buffer/QD-interface and less affected by distortions. More details on the accuracy of CELFA and sources of error can be found in [23]. Scanning transmission electron microscopy (STEM) with a high-angle annular dark-field (HAADF) detector was carried out using a ZEISS 922 Omega with a LaB$_6$ emitter to obtain composition sensitive (Z contrast) images.

Low temperature (5 K) PL spectra were acquired using an InGaAs detector and a spectrometer equipped with a 600 mm$^{-1}$ grating. The excitation was carried out by the 442 nm and 325 nm lines of a HeCd laser. The signal was amplified by a standard lock-in technique.

3. Results and discussion

3.1. Influence of the InAs deposition rate on the morphology of InGaAs QD structures and distribution of group-III elements

The optimization of the parameters substrate temperature, deposited amount of InAs and growth rate is essential with respect to achieving 1.3 and 1.5 µm luminescence from InAs QD structures. We focus here on the effect of the InAs deposition rate for otherwise already optimized conditions (see §2 and [9]) which needs to be reduced to extremely low values. A strong effect is observed on the PL peak position which shifts from 1.21 eV for sample $S_{\text{high}}$ (0.08 ML/s) to 1.04 eV for $S_{\text{low}}$ (0.0056 ML/s) at 6 K. Bright-field (BF) TEM micrographs of plan-view samples are presented in figure 1(a) for $S_{\text{low}}$ and in 1(b) for $S_{\text{high}}$. The growth-rate reduction is correlated with a significant decrease of the QD density from $1.1 \times 10^{11}$ cm$^{-2}$ to $2.0 \times 10^{10}$ cm$^{-2}$ and the strain field associated with the QDs changes from a small dot-like shape for $S_{\text{high}}$ to a square-shaped one with a larger extension for $S_{\text{low}}$. 
**Figure 1.** Plan-view BF images taken along the [001]-zone axis of samples with an InAs deposition rate of (a) 0.0056 ML/s and (b) 0.08 ML/s. The magnification in (a) and (b) is identical.

**Figure 2.** Colour-coded cross-section maps of the In distribution in the samples (a) S_{low} and (b) S_{high}. Note the different scaling of the colour coding for the In concentration in (a) and (b). The arrows mark WL regions where the composition profiles in figures 3(a,b) are extracted.

**Figure 3.** In-concentration profiles as a function of distance in growth direction in the WL regions indicated by arrows in figures 2(a,b) for samples (a) S_{low} and (b) S_{high}. Dashed lines are fit curves according to Eqs. (1) and [11].

Colour-coded cross-section views of the In distribution obtained by CELFA are presented in figure 2(a) for S_{low} and in 2(b) for S_{high}. Note that a different scaling of the colour coding was used. The QDs with a height of about 10 nm clearly emerge from the WL in the low-growth-rate sample. The In
concentration in the QD in $S_{\text{low}}$ increases gradually to about 60% at the top of the QD. However, the real In concentration will be higher because we measure average values given by the composition of the QD and the embedding GaAs cap layer. In contrast, Figure 2(b) shows a layer with fluctuating In concentration up to only 30% with a slightly varying thickness around 6 nm for $S_{\text{high}}$. The regions with high In concentration are assigned to the QDs but the interpretation of figure 2(b) is not straightforward because the high QD density has to be considered with respect to the finite TEM sample thickness. QDs may therefore overlap along the electron-beam direction. However, it is clear that the height of the QDs is rather small in $S_{\text{high}}$ and the QDs are almost embedded completely in the WL. The apparent high In concentration (light blue colour) in the buffer and cap layers above and below the QDs is an artefact induced by the strong bending of the (002) planes in the vicinity of the QDs.

Composition profiles along the [001]-growth direction are presented for $S_{\text{low}}$ and $S_{\text{high}}$ in figure 3. The profiles are measured in the planar WL regions indicated by arrows in figures 2(a,b). The asymmetry of the profiles is a characteristic feature of In segregation. Maximum In concentrations between 20 and 25% are found independently of the deposition rate. The total amount of indium in the WL corresponds to 1.7 ML in both samples which is obtained by the integration of the composition profiles.

The measured In concentrations $x$ in figure 3 were fitted by applying the segregation model of Muraki [11] using the equations

$$x(n) = \begin{cases} 0 & : n < 1 \\ x_0(1 - R^n) & : 1 \leq n \leq N \\ x_0(1 - R^n)R^{n-N} & : n > N \end{cases}$$

where $n$ is the ML number in growth direction, $x_0$ the nominal In concentration, $R$ the segregation efficiency and $N$ the total amount of deposited indium expressed in MLs of InGaAs. The parameters $x_0$, $N$ and $R$ are considered as fit parameters. As these parameters define different characteristics of the profile ($x_0$ and $R$ the slope of the increasing part and the maximum value, $R$ the shape of the decreasing part, and $N$ the position of the maximum), unique values of the fit parameters are obtained. From several fitted profiles in both samples, In-segregation efficiencies of $0.69 \pm 0.03$ for $S_{\text{low}}$ and $0.7 \pm 0.03$ for $S_{\text{high}}$ are derived which agree within the error margins.

The evaluation of the concentration profiles demonstrates that the properties of the WL and the segregation efficiency do not depend on the InAs deposition rate. The change of the island density and size results solely from the redistribution of deposited InAs exceeding 1.7 ML. The slow growth promotes In redistribution which leads to an accumulation of indium in large QDs. Fast surface diffusion is necessary for the redistribution process which limits $T_{\text{sub}}$ to relatively high values around 500 °C. However, to model quantitatively the PL energy, the real In concentration in the QDs must be known. It is in particular interesting to see if binary InAs is contained in the top region of the QDs. A procedure is presented in the next subsection which takes into account the embedding GaAs cap layer.

Considering the substrate temperature of 500 °C during InAs deposition, the measured segregation efficiency of 0.7 is low compared to values compiled in [13] which can be attributed to the high BEPR of 80 applied during the growth of these samples. Using a lower substrate temperature of 475 °C and a BEPR of only 4, Muraki et al. [11] found a high segregation efficiency of 0.83 which underlines the strong effect of the BEPR on In segregation.

3.2. Group-III element distribution in InGaAs quantum dots

The determination of the real composition of embedded QDs requires – in addition to the CELFA data – information on the local sample thickness as well as shape and size of the QDs. The following analysis was carried out for QDs in sample $S_{\text{low}}$ because the QDs in $S_{\text{high}}$ are too small. The local sample thickness was determined by recording a tilt series of (002) dark-field images. The sample was tilted around an axis parallel to the [100] direction in steps of 5° starting close to the [010]-zone axis. With increasing tilt angle the projection of the thin WL broadens. By measuring the width of the projected WL at a given tilt angle the local sample thickness can be calculated using simple
trigonometry in analogy to the procedure described in [16]. Images taken at large tilt angles also show whether a QD is contained completely in the TEM sample or whether it is sectioned by the sample surface. The latter would be useless for further study because the measured In concentration would always be too low.

The square symmetry of the QD strain field in figure 1(a) suggests a pyramidal island structure with edges aligned along the [100] and [010] directions. This shape is verified by HAADF STEM cross-section images as shown in figure 4(a) where a pyramidal-type island with a truncated top is seen. Angles of about 45° between the side faces and base suggest \{101\} side faces.

![Figure 4.](image)

**Figure 4.** (a) HAADF STEM cross-section image of an InGaAs island and colour-coded cross-section maps of the In-distribution obtained by CELFA (b) before correction and (c) after correction. Note the different scaling of the colour coding in (b) and (c).

A colour-coded map of the In distribution in a QD before processing is presented in figure 4(b) which shows a maximum In concentration of 46 % in the center of the QD. To minimize artefacts due to lattice-plane bending (200) lattice-fringe images were evaluated. To correct the CELFA data for the thickness-dependent averaging effect, a projection of an \{101\}-facetted pyramid was fitted on the QD in figure 4(b). The relevant parameters for the correction are the sample thickness \(d = 32 \text{ nm}\) (in this case) and the distance \(d_s\) between the \{101\} facets at the vertical position \(z_s\). The ratio \(V\) between the sample thickness \(d\) and the local thickness of the QD \(d_z\) (see figure 4(b)) is given as a function of the vertical coordinate \(z_s\), \(V(z_s) = d / (d_B + 2d_B - z_s)\) with the basis width of the pyramid \(d_B\) at the position \(z_B\). Multiplication of \(V\) with the In concentration obtained by CELFA yields the corrected In concentration displayed in figure 4(c) (note the different scale of the colour coding in figures 4(b,c)).

A maximum In concentration of 95 % is reached at the top of the island. The thickness correction also leads to a rearrangement of the In distribution with the highest In concentration in the top region of the QD as opposed to the uncorrected data. However, a large fraction of the QD still contains significantly lower In concentrations starting at about 50 % near the base. This is in rather good agreement with earlier investigations on similar QDs by different methods like electron energy loss spectroscopy by Wang et al. [18], who used a similar procedure for the correction of the measured In concentration, and cross-sectional scanning tunnelling microscopy [19].

There are several factors which affect the accuracy of the data. First, the QD shape may deviate from the ideal shape of an \{101\}-facetted pyramid which applies especially to the top of the QD which is not completely flat. Second, a non-quadratic QD base cannot be detected in cross-section samples and would lead to an error for the corrected In concentrations. Another contribution to the error is caused by the thickness measurement because the boundaries of the projected WL are blurred, limiting the accuracy of the width measurement and the derived sample thickness. Apart from the intrinsic errors of CELFA [23], the In concentration should be measurable with a relative error of about 10 % using the suggested procedure with a more precisely known sample thickness and neglecting the fact that shape information along the electron-beam direction for a specific QD is not accessible.
3.3. Influence of the substrate temperature during GaAs cap-layer growth

In the following we present results which illustrate the effect of the reduction of the substrate temperature during GaAs cap layer deposition. The investigation of the plan-view samples (not presented here) shows that the sample capped at 485 °C contains only defect-free islands while a small density of relaxed islands with defects is observed for the sample capped at 410 °C. The formation of relaxed islands can be attributed to In redistribution during the growth interruption after InAs deposition that was necessary to reduce the substrate temperature. Figures 5(a-c) show colour-coded maps of the local In concentration for the samples overgrown at $T_{\text{cap}} = 485$ °C (sample $S_{485}$), $T_{\text{cap}} = 410$ °C (sample $S_{410}$) and $T_{\text{cap}} = 350$ °C (sample $S_{350}$). Again, different colour scales were chosen in figures 5(a-c) for a more differentiated visualization of the In distribution. In-concentration profiles along the [001]-growth direction in the WL regions (at the position of white arrows in figures 5(a-c)) of all samples are displayed in figures 5(d-f).

According to figure 5 the reduction of $T_{\text{cap}}$ has a strong effect on the In distribution. Lowering $T_{\text{cap}}$ from 485 °C to 410 °C leads to a decrease of the thickness of the WL with a full width at half maximum (FWHM) for sample $S_{485}$ of 10 ML to 5 ML for $S_{410}$. The reduction of the WL width is compensated by a corresponding increase of In concentrations with maximum values in the WL of 13% in $S_{485}$ and 27% in $S_{410}$ (figures 5(d,e)). These effects are a clear indication of the reduction of In segregation.

Sample $S_{350}$ does not show the expected behaviour (figures 5(c,f)) because a strong broadening of the In distribution (WL with a FWHM of 13 ML) and a general reduction of the In concentration is observed with an overall maximum In concentration of only 14%. We assume that interdiffusion occurred in this sample for the following reasons. A high concentration of point defects was probably introduced due to the low $T_{\text{cap}}$ which is supported by the weak PL intensity of this sample. A potentially high vacancy concentration may lead to strong interdiffusion of In and Ga atoms after increasing $T_{\text{cap}}$ from 350 °C after the deposition of 20 ML GaAs to 570 °C. The dominant influence of interdiffusion on the In distribution is also supported by the symmetrical composition profile in figure 5(f) in contrast to the asymmetrical ones for $S_{485}$ and $S_{410}$.

Segregation efficiencies were determined on the basis of the measured profiles using Eqs. (1). The fit curves (dashed lines) are included in figures 5(d,e). We obtain $R = 0.76 \pm 0.02$ for sample $S_{485}$ which exceeds $R = 0.7$ obtained for the samples in §3.1 deposited at 500 °C. However, here a low V:III BEPR of only 10 was used. For sample $S_{410}$ $R = 0.59 \pm 0.09$ is derived. This value with a large error has to be considered with caution because the temperature was changed during the growth. It was 485°C for the ascending part of the profile during InAs deposition and 410 °C for the descending part associated with the cap-layer growth. The amount of InAs in the WL is 1.7 – 1.8 ML in all samples in agreement with the values obtained for the samples in §3.1.

Lowering $T_{\text{cap}}$ to 410 °C leads to an obvious reduction of segregation-induced intermixing which could indeed be utilized as a technique to maximize the indium content in QDs which are grown under optimized conditions for long-wavelength luminescence.
Figure 5. Colour-coded cross-section maps of the In distribution obtained by CELFA for samples (a) $S_{485}$, (b) $S_{410}$ and (c) $S_{350}$ and corresponding In-concentration profiles extracted from the WL regions marked by the arrows in the composition maps for samples (d) $S_{485}$, (e) $S_{410}$ and (f) $S_{350}$.

4. Summary
A TEM and PL study of MBE-grown InAs QD structures was performed with respect to obtaining large QDs with a high In concentration which are required for luminescence in the 1.3 $\mu$m regime. Special attention was paid to In segregation which strongly affects the distribution of group-III elements in these structures. In-segregation coefficients were derived from atomic-scale composition profiles obtained on the basis of $\{200\}$ lattice-fringe HRTEM images by applying the CELFA evaluation technique.

The effect of the InAs deposition rate was studied under otherwise optimized conditions for large QDs. A high substrate temperature of 500 $^\circ$C and a low growth rate of 0.0056 ML/s are essential to allow In redistribution during the deposition process. The In-segregation efficiency of 0.7 is low compared to literature data which is attributed to the high V:III BEPR of 80 which is – in addition to
the temperature – an important parameter for In segregation. The same In-segregation efficiency was observed for a deposition rate of 0.08 ML/s. A procedure was developed for the determination of the real In concentration in InAs QDs which takes into account the finite TEM sample thickness and the embedding of the QDs in a GaAs cap layer. The analyses were carried out for QDs grown under optimized conditions for long-wavelength luminescence. It is found after correction of the measured In distribution that the In concentration increases from about 50 % at the bottom to almost 100 % at the top of the QD.

Finally, the effect of the temperature during cap-layer growth was studied. The InAs QD layers were deposited under identical conditions but the GaAs capping was performed at 485, 410 and 350 °C. The In concentration in the QD structure increases strongly due to the reduction of In segregation during cap-layer growth at 410 °C. A further decrease of $T_{\text{cap}}$ to 350 °C leads to strong broadening and intermixture between InAs and GaAs. This effect is attributed to interdiffusion due to a high vacancy concentration introduced at 350 °C and the increase of the temperature to 570 °C after the deposition of 20 ML GaAs. Nevertheless, lowering of the substrate temperature during cap-layer growth can be a reasonable procedure to maximize the In concentration in InAs QDs.

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