

Quantification of segregation and strain effects in InAs/GaAs quantum dot growth

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Reflection high-energy electron diffraction measurements of the critical thickness θ_{crit} for quantum dot (QD) formation have been used to quantify the effects of indium segregation and strain on the growth of bilayer InAs/GaAs(001) QD structures. These are not straightforward to deconvolute, because of the complex issues that arise during the growth and capping of the QDs. Segregation and out diffusion of In from buried QDs are shown to occur for GaAs thicknesses up to ~ 6 nm at 580 °C. The existence of a floating In adlayer on the surface of the GaAs-capping layer as a result of In segregation is apparent at much lower substrate temperatures (510 °C). The relative contribution of both segregation and strain on the reduction of θ_{crit} during the growth of a second InAs layer is assessed. Compared with segregation, strain from the buried QDs can be measured through significantly larger capping thicknesses (~ 30 nm) under these conditions. © 2005 American Institute of Physics. [DOI: 10.1063/1.2133904]

I. INTRODUCTION

III-V semiconductor quantum dots (QD) look promising candidates as the active region in next generation optoelectronic devices, such as low-chirp lasers,¹ semiconductor optical amplifiers,² and single-photon emitters.³ The InAs/GaAs(001) QD system is of particular technological interest. Recent studies have shown that multilayer QDs can be used to extend the emission wavelength beyond 1.3 μm , with 1.5 μm emitters being demonstrated.⁴ Multilayer QD structures are generally employed for in-plane laser diodes and it is now well established that their growth and properties can be complex, in particular, when the QD layers are separated by relatively small GaAs spacer layers (typically ≤ 20 nm). Effects such as strain propagation through the spacer layer,⁵ In segregation,^{6–8} and In/Ga intermixing during the various growth steps^{4,9–12} all have a strong influence on the optical and structural properties but are still not fully understood. One important aspect is the surface morphology of a single layer of GaAs-capped QDs. It is well established that surface undulations are produced during GaAs capping of the QDs at the QD growth temperature and these prevail even for relatively large capping thicknesses.^{13,14} During annealing at 580 °C, however, an atomically flat surface is produced^{15,16} and this rules out any possible influence of surface curvature on the growth of a consecutive layer of QDs.

In this paper we focus on the processes that lead to changes in the critical thickness (θ_{crit}) for QD formation in bilayer structures, as measured by *in situ* reflection high-energy electron diffraction (RHEED). The study of different bilayer structures, in which the first layer is either a pure

two-dimensional (2D) layer of InAs or an InAs QD layer, provides a method for deconvoluting segregation and strain effects and allows direct quantification of In segregation during multilayer QD growth. The results have important implications for the growth of multilayer InAs/GaAs QD-based devices.

II. EXPERIMENTAL DETAILS

All samples were grown on epi-ready GaAs(001) substrates (n^+ , Si doped) in a molecular-beam epitaxy (MBE) system (DCA Instruments) equipped with RHEED. The InAs growth rate [~ 0.015 monolayer per second (ML s^{-1})] was carefully controlled by growing a single QD layer at the temperature of the $(2 \times 4) \rightarrow c(4 \times 4)$ surface reconstruction change (510 °C), as monitored by RHEED. The growth mode transition, which is monitored by the abrupt appearance of chevrons in the $[\bar{1}10]$ azimuth of the RHEED pattern, always occurred at 130 ± 2 s ($\theta_{\text{crit}} = 2$ ML) for these growth conditions. More details of the growth process can be found elsewhere.⁴ Photoluminescence (PL) measurements on the capped QD samples were performed using an Ar⁺ laser dispersing the light with a SPEX 0.5 m monochromator and detected with a cooled Ge diode.

III. RESULTS AND DISCUSSION

Figure 1 shows the variation of the second layer critical thickness ($\theta_{\text{crit},2}$) as a function of spacer layer thickness (S) for a series of QD bilayer structures, in which the first layer consists of a QD layer (2.5 ML InAs; average height of the QDs as measured by atomic force microscopy (AFM) = 5.9 nm) grown at 510 °C. The full triangles correspond to samples that were annealed at 580 °C after GaAs capping of the first QD layer and prior to growth of the second QD layer, for which $\theta_{\text{crit},2}$ was then recorded. The empty triangles show how $\theta_{\text{crit},2}$ varies for samples in which no growth in-

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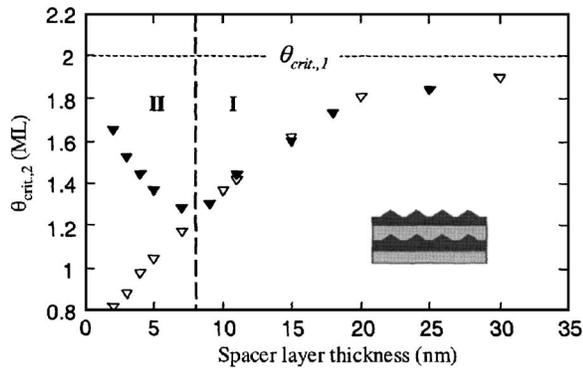


FIG. 1. Influence of spacer layer thickness on the critical thickness for QD formation in the second layer above a buried QD layer ($\theta_{\text{crit},2}$). In some cases the GaAs spacer layer was annealed at 580 °C for 10 min (full triangles) and in others no growth interruption was performed prior to deposition of the second layer (clear triangles). The distinction between the two regimes is indicated by the dashed vertical line at $S \sim 8$ nm. The dashed horizontal line shows the measured $\theta_{\text{crit},1}$ value for comparison.

ruption and annealing were performed prior to second layer QD growth. The dashed horizontal line shows $\theta_{\text{crit},1}$ (2 ML) for easier comparison.

For spacer layer thicknesses up to ~ 30 nm there is a significant reduction in $\theta_{\text{crit},2}$ compared with $\theta_{\text{crit},1}$. An extrapolation of the data suggests that $\theta_{\text{crit},2} = \theta_{\text{crit},1}$ when $S \sim 50$ nm. It is not possible to distinguish between the two series of samples for $S \geq 8$ nm (regime I). However, for $S < 8$ nm (regime II), there is a significant difference between the two types of samples. $\theta_{\text{crit},2}$ for the nonannealed samples decreases further, whereas in the case of the annealed samples, $\theta_{\text{crit},2}$ reaches a minimum for $S = 7$ nm before increasing again at even smaller values of S .

Strain fields are known to have a significant impact on $\theta_{\text{crit},2}$ in bilayer QD samples and it has been established that this effect can propagate up to ~ 40 nm, with this distance depending on the size of the first layer QDs.¹⁷ The strain fields are the result of the (elastic) lattice relaxation of the dots in the first layer¹⁸ and can be rationalized in terms of a local expansion of the GaAs lattice above the QDs. This lattice expansion propagates vertically through the spacer layer, and the lattice constant of the GaAs matrix on the surface above a buried dot is therefore larger than the value for the unstrained case (5.65 Å). This strain-energy modulation leads to enhanced migration of second layer In adatoms towards the areas with increased lattice constant,¹⁹ and the locally increased surface population of In adatoms leads to a reduction in $\theta_{\text{crit},2}$.

The similarity of the $\theta_{\text{crit},2}$ values for the annealed and nonannealed structures in regime I suggests that morphological features of the GaAs spacer layer do not affect second layer QD nucleation.

For $S < 8$ nm (regime II) the $\theta_{\text{crit},2}$ values vary depending on whether the samples have been annealed or not. This variation is a combination of two factors, both a result of InAs desorption during annealing for the samples that are only covered with a very thin GaAs layer. The first effect is based on the existence of floating In adatoms on the surface of the GaAs spacer layer and will be discussed in Sec. III A.

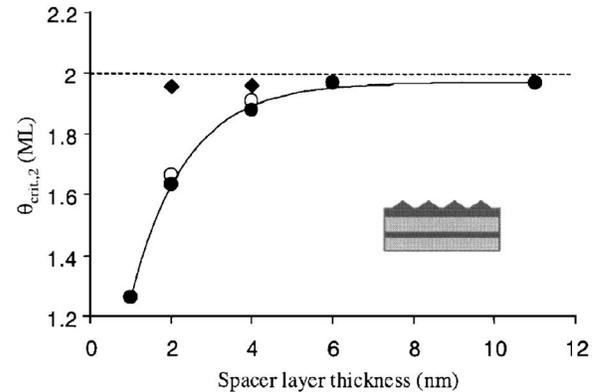


FIG. 2. Influence of spacer layer thickness (S) on the critical thickness $\theta_{\text{crit},2}$ of bilayer samples with the sequence of 2D InAs layer (1.75 ML)/spacer layer/QD layer (full circles). The diamonds (clear circles) show samples that were annealed for 10 min at 580 °C (510 °C) after growth of the spacer layer. The line is based on a fit of the In segregation efficiency. The dashed horizontal line shows the value of $\theta_{\text{crit},1}$ for an isolated single QD layer grown at 510 °C.

The second effect is based on segregation of In from the buried QDs through the GaAs matrix and will be discussed in Sec. III B.

A. Surface segregation

The presence of a floating In adlayer on the surface of the GaAs cap is a result of surface exchange of In with Ga adatoms during the growth of the GaAs spacer layer. These In adatoms desorb during annealing at high temperature, causing $\theta_{\text{crit},2}$ to increase for low values of S compared with the nonannealed samples (Fig. 1). However, an estimate of the magnitude that this effect alone should have on the differences in $\theta_{\text{crit},2}$ in regime II shows that it cannot fully explain the drastic changes observed. This estimate is based on results obtained from a series of samples which are shown in Fig. 2. It shows how $\theta_{\text{crit},2}$ varies as a function of S for a series of different QD samples in which the basic structure comprised an InAs 2D layer with a thickness of 1.75 ML (i.e., $< \theta_{\text{crit},1}$) grown at 510 °C, followed by a GaAs spacer layer of varying thickness S . The second InAs layer was then deposited without any growth interruption and $\theta_{\text{crit},2}$ was recorded (full circles). There is a significant drop in $\theta_{\text{crit},2}$ as S is reduced below 6 nm (~ 21 ML). This reduction is due to the existence of surface In adatoms as a consequence of segregation through the GaAs spacer layer during growth. The thinner the GaAs spacer layer, the higher the concentration of surface In adatoms, and the more pronounced the reduction in $\theta_{\text{crit},2}$. The thermodynamic driving force for the floating of In on GaAs has been estimated by Moison *et al.*⁶ and is a result of the stronger bonds in GaAs compared with InAs. It is interesting to note that in contrast to the InAs-on-GaAs interface, the GaAs-on-InAs interface is not atomically sharp. Unlike the case of buried QDs, a buried 2D layer of InAs does not produce inhomogeneous strain fields, because it grows pseudomorphically. Strain interactions can therefore be ruled out.

The process of In floating on a 2D GaAs layer deposited at 510 °C and subsequent In desorption during annealing at 580 °C are shown schematically in [Fig. 3(a)]. The floating

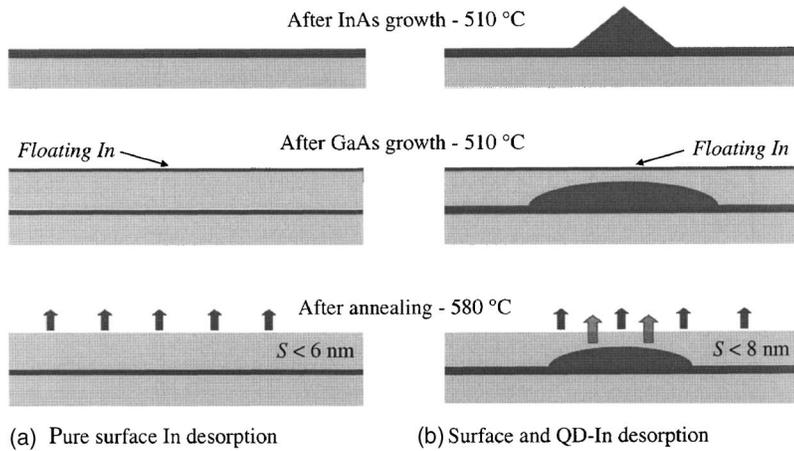


FIG. 3. (a) Schematic of the In floating layer and subsequent desorption in the case of a pure 2D InAs layer overgrown with GaAs. (b) Schematic showing both the presence of an In floating layer as well as In vertical segregation.

behavior can be confirmed by close observation of the RHEED patterns. After deposition of 2 nm GaAs, the RHEED pattern along the $[\bar{1}10]$ azimuth shows a (1×3) reconstruction, indicative of an $\text{In}_x\text{Ga}_{1-x}\text{As}$ alloy.²⁰ For $S = 4$ nm, the RHEED pattern reveals a mixed phase, with the (1×3) reconstruction dominating, although the existence of a $c(4 \times 4)$ reconstruction indicates some domains of pure GaAs.²¹ For $S = 6$ nm, the situation changes and the $c(4 \times 4)$ pattern dominates, although the (1×3) reconstruction can still be observed. The RHEED pattern is essentially pure $c(4 \times 4)$ for $S = 8$ nm, indicating that the surface In concentration is negligible and it is reasonable to conclude that the segregation of In atoms through the growing GaAs layer occurs up to a maximum thickness of ~ 8 nm for these growth conditions.

The effects on $\theta_{\text{crit},2}$ of the introduction of a growth interruption and annealing step can also be seen in Fig. 2. For the samples with $S = 2$ and 4 nm, a growth interruption of 10 min was performed before depositing the second InAs layer. The clear circles represent an interruption with unchanged temperature (i.e., 510 °C) whereas the diamonds correspond to samples which were annealed at 580 °C before second layer deposition. Annealing at 510 °C before second layer InAs deposition does not change the RHEED pattern, regardless of the value of S , and the rate of In desorption is clearly very low at this temperature. For this reason $\theta_{\text{crit},2}$ in the second layer remains unchanged. During annealing at higher temperatures (580 °C), however, the RHEED pattern changes in all cases within 1–2 min into a (2×4) pattern, the stable surface reconstruction for GaAs(001) at this temperature.²¹ This change is due to the thermal desorption of InAs from the surface and explains why the growth of the upper QD layer after annealing at higher temperatures is similar to the growth of an isolated QD layer (see $\theta_{\text{crit},1}$ which is shown as a dashed horizontal line in Fig. 2).

An estimate of the surface segregation efficiency can be obtained using a simple model originally developed by Toyoshima *et al.*²² for InGaAs growth on GaAs(001). If the fraction of In which segregates through each ML of GaAs during deposition is defined by R , then the amount of segregated InAs in the top layer (X_n) is given by $X_n = X_0 R^n$, where X_0 is the amount of free InAs (in ML) on the starting surface

when GaAs growth is initiated, and n is the number of deposited GaAs monolayers. The best fit to the experimental data is shown as the solid line in Fig. 2 with $X_0 = 1.46$ ML and $R = 0.814$. It should be noted that X_0 is significantly different from the total amount of InAs deposited (1.75 ML). This discrepancy can be rationalized by assuming that only a fraction of the deposited In is incorporated as InAs (0.29 ML), the rest remaining afloat on the surface. The floating In is then incorporated progressively during the first 8 nm of GaAs capping. It should be emphasized that this simple model is based on a step by step and not a continuous flow mechanism. Strictly speaking the model is only valid for $n \geq 2$, otherwise the amount of surface In in the top layer would be > 1 ML (for $n < 2$).

It is interesting to note that the In segregation ratio ($R = 0.814$) is close to the one determined by Toyoshima *et al.* (0.81–0.85), who performed growth at the slightly higher temperature of 520 °C.²² This suggests that segregation of In is qualitatively and quantitatively similar for overgrowth of InAs with GaAs and the deposition of InGaAs on GaAs. Considering the higher In flux used in the work of Toyoshima *et al.* (0.09 ML s^{-1} compared with 0.015 ML s^{-1} in our work) this means that the segregation process is controlled thermodynamically under these conditions.

B. Vertical In segregation

The buried first layer dots clearly act as a second source of In that can desorb during annealing of the GaAs spacer layer. These dots are only protected by a thin (In)GaAs layer and may be subject to partial In desorption during annealing. However, in this case the process of In loss is due to diffusion through the (In)GaAs-capping layer above the QDs and is *not* a pure surface process. In the following we use the expression *vertical In segregation* in order to avoid confusion. As a result of In desorption, the strain associated with the buried dots becomes weaker and the influence of strain on second layer QD nucleation diminishes, causing $\theta_{\text{crit},2}$ to return to the value of an isolated layer (~ 2 ML). Both effects, the loss of In from the surface and from the QDs, are shown schematically in Fig. 3(b). They contribute to the increase in $\theta_{\text{crit},2}$ when thin GaAs spacer layers are annealed at higher temperatures (see full triangles in Fig. 1, regime II).

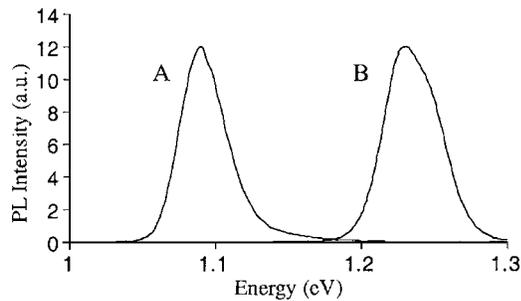


FIG. 4. Normalized, low excitation PL spectra (0.1 W cm^{-2}) of two single layer QD samples recorded at 10 K. Spectrum A shows ground-state emission from a reference sample grown under standard conditions. Spectrum B has been recorded from a sample in which after an initial GaAs cap of 3 nm the sample was annealed at 580°C for 10 min, followed by deposition of a further 100 nm of GaAs.

The partial desorption of material from the buried dots during annealing at high temperatures can be directly confirmed by optical measurements. The QD size, composition, and strain state all have a pronounced effect on the emission wavelength.⁴ Partial desorption of material from the buried QDs during annealing results in a decrease in the total In content of the dots, causing a decrease in size and/or indium composition. A blueshift in the PL spectrum of these dots should therefore be observed.

The normalized, low-temperature, low excitation PL spectrum of an isolated single layer of QDs, grown under standard conditions, is shown in Fig. 4 (spectrum A). The QDs were emitted at an energy of 1.09 eV with a full width at half maximum (FWHM) of ~ 33 meV. A similar structure was grown but the QDs were capped with 3 nm of GaAs, followed by the annealing treatment at 580°C and the deposition of a further 100 nm of GaAs. The emission properties from this sample are strongly altered (spectrum B) compared with A. The slightly broader emission (FWHM > 45 meV) now occurs at an energy of 1.23 eV. The blueshift of ~ 140 meV is a direct consequence of the loss of material from the dots during annealing, although no quantitative estimate can be made about the reduction in QD size or indium composition. The concomitant enhancement of the linewidth is also an indication that the QD structure has been altered by annealing, introducing more fluctuations in the composition and size of the QD ensemble. Both observations confirm the suggestions regarding InAs desorption from the first layer dots during annealing for small S . This desorption is eventually responsible for the reduced strain induced by the first QD layer in the second layer and therefore for the observed increase in $\theta_{\text{crit},2}$. It should be noted that the PL spectrum for an identical single QD layer, but with an annealed cap layer of 10 nm as opposed to 3 nm, leads to an essentially identical spectrum to that shown for A. The QDs are therefore fully protected by a 10 nm cap and annealing at 580°C does not lead to measurable In desorption.

C. Discussion

In an attempt to *quantitatively* compare the effects of the two In–Ga (ad)atom exchange processes that are responsible for the floating In (surface exchange during growth) and ver-

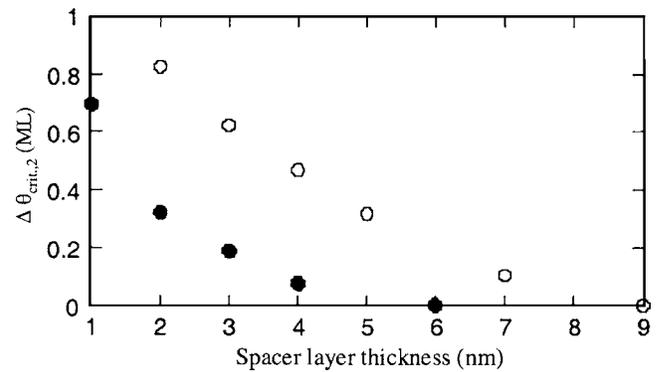


FIG. 5. Diagram presenting the contribution, respectively, of the pure surface In adatom segregation (full circles) and the sum of the contribution of both surface and bulk In segregations (clear circles) on the reduction of the critical thickness in the second layer above an InAs layer. The values corresponding to the full circles have been obtained from those of Fig. 2, whereas the clear circles denote the difference between the values in Fig. 1.

tical In segregation from the buried dots, respectively, the difference between the two $\theta_{\text{crit},2}$ values (nonannealed versus annealed samples) from regime II of Fig. 1 is plotted against S (see clear circles in Fig. 5). The $\Delta\theta_{\text{crit},2}$ values correspond to the combined effects of surface exchange and vertical segregation of In (ad)atoms on the relative increase in $\theta_{\text{crit},2}$ when the spacer layer is annealed at 580°C . Also plotted in Fig. 5 are the values of $\Delta\theta_{\text{crit},2}$ for the two series of samples in which the buried layer of QDs was replaced by a 2D InAs layer (see Fig. 2). The reduction in $\theta_{\text{crit},2}$ from the annealed to the nonannealed samples is shown as the full circles in Fig. 5. In contrast to the structures containing a buried layer of QDs, no effects of the possible loss of In from the buried layer during annealing on $\theta_{\text{crit},2}$ are expected, because the pseudomorphic 2D InAs layer does not produce strain fields. Therefore, the full circles are the pure contribution of surface In/Ga exchange on the reduction of $\theta_{\text{crit},2}$ and they correspond to the amount of floating In.

The difference between the clear and full circles in Fig. 5 corresponds to the contribution of In desorption from the buried dots on the reduced critical thickness for QD formation in the second layer. However, despite the ease of quantifying the floating In process, no quantitative estimate can be made about the amount of In lost due to diffusion from the QDs, *only about its effect on the critical thickness*. This effect is indirect and a consequence of the reduced strength of the strain from the buried dots due to InAs desorption.

These results clearly show that strain from the buried QDs plays the major role in influencing the process of QD formation in the second layer and this effect operates up to a GaAs thickness of at least 30 nm. Indium floating generally has a minor effect on the growth of the second InAs layer and its effect can only be measured for spacer layer thicknesses of up to ~ 6 nm.

In this context the difference between In floating during GaAs deposition, which is a surface exchange process, and vertical In segregation during annealing has to be discussed. Desorption of InAs from the buried dots is qualitatively a different process. It is characterized by the diffusion of In atoms through a thin (In)GaAs layer. The resulting kinetic limitation is therefore significantly more pronounced com-

pared with the process of surface segregation, in which the exchange between Ga and In adatoms occurs in the upmost layer (of $\text{In}_x\text{Ga}_{1-x}\text{As}$). Similar to the Ge/Si system²³ the thermal energy necessary for activation of bulk diffusion is much higher.²⁴ Indeed, the floating In has been estimated to occur up to a GaAs thickness of 6 nm, but the substrate temperature in this case was low (510 °C). InAs desorption from the buried dots, however, only occurs at a much higher temperature. At similar nominal GaAs spacer layer thicknesses, the threshold for the thermal activation of the two processes is therefore very different.

Furthermore, the deviation between the nominal and effective GaAs thicknesses on top of the QDs has to be taken into account. The real thickness of the protective GaAs layer directly on top of the dots is significantly smaller than the nominal thickness of the deposited layer, because the effective GaAs growth rate above the QDs is initially lower than the growth rate in the surrounding parts of the dots.¹³ As a result, the effective GaAs layer directly above a dot is probably extremely thin in regime II and vertical In adatom segregation will occur, provided that the substrate temperature is sufficiently high.

An additional factor that has to be taken into account regarding the proposed out diffusion of In is the tensile strain above a buried dot. The lattice constant of the GaAs (or the $\text{In}_x\text{Ga}_{1-x}\text{As}$ alloy) above a dot is slightly increased from that of pure GaAs.¹⁸ One might speculate that this difference may facilitate the exchange of In and Ga adatoms compared with the case of unstrained GaAs.

IV. CONCLUSION

The reduction of the critical thickness for QD formation in the second layer above a buried InAs layer is a combination of two effects. The first is the segregation of In through the GaAs spacer layer during growth, which leads to the presence of floating In adatoms. Indium is therefore present on the surface before deposition of the second InAs layer, and the amount of InAs required to reach the 2D → 3D growth mode change is reduced. This effect only operates for GaAs layers of a thickness of less than ~6 nm at a growth temperature of 510 °C. It can be compensated for if a high-temperature (580 °C) annealing treatment is applied before growth of the second layer. The second and more pronounced effect for the reduced critical thickness in the second layer is a result from the strain fields created by the buried QDs. It generally dominates over segregation and operates up to a GaAs thickness of 30 nm for both annealed and nonannealed structures. For very small spacer layers, however, ($S < 8$ nm), the GaAs does not protect the dots

from the first layer completely, thus leading to significant diffusion of In through the GaAs and consequently to In desorption during annealing at 580 °C. This effectively leads to a reduction in the size/composition of the buried QDs from the first layer. In turn, the strain fields are weaker and therefore the critical thickness measured from the dots in the second layer increases again. Not surprisingly, if no annealing procedure for thin GaAs spacer layers is applied, the critical thickness decreases even more.

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