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Optical properties of low-strained $In_xGa_{1-x}As/GaAs$ quantum dot structures at the two-dimensional–three-dimensional growth transition

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$In_xGa_{1-x}As/GaAs$ quantum dots (QDs) were grown by solid source molecular beam epitaxy for indium contents of around 30%, which assures the QD growth in the very low strain limit. The structures were fabricated for a constant nominal $In_xGa_{1-x}As$ layer thickness but varying content (strain) from below to far above the critical thickness conditions, which has allowed to detect the onset of three-dimensional island formation and their evolution with the increasing material amount (for higher In contents the critical thickness for island formation is smaller and hence a larger fraction of the $In_xGa_{1-x}As$ layer is spent on dot formation). In order to investigate the properties of such an uncommon QD system, photoreflectance and photoluminescence, combined with scanning electron microscopy, have been used. Optical transitions connected with the ternary layer have been observed and followed from the lowest content quantum well case through the transformation into three-dimensional islands on the wetting layer (WL) and a coexistence of the QD-related and WL-related transitions. Due to the observation of both heavy hole and light hole related transitions in photoreflectance spectra, the thickness of the wetting layer versus changed indium content could be determined, comparing the experimental data with the results of the effective mass envelope function calculations. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2208296]

INTRODUCTION

Self-assembled semiconductor quantum dots (QDs) have attracted considerable interest during recent years. Many important fundamental properties related to three-dimensional carrier confinement, deltalike density of states, and atomlike properties have been discussed in the literature and a lot of practical applications have been proposed, from more common ones, such as those in lasers and detectors,[1–3] to very sophisticated ones, such as single electron transistors,[4,5] single photon sources,[6] or quantum bits.[7,8] Independently of the material system used, the self-assembled QD growth is driven by a very large lattice mismatch between the QD layer and the substrate, causing typically a formation of three-dimensional (3D) islands on a thin two-dimensional (2D) layer [called wetting layer (WL)], i.e., a process known as the Stranski-Krastanov growth mode.[9] When the strain is reduced by decreasing the In content in the deposited layer, it becomes difficult to self-create 3D islands in favor of planar 2D growth. On the other hand, such structures offer interesting properties and many possible applications mainly driven by the QD shape engineering. Therefore, this kind of growth has been a challenge for semiconductor technology in the last years. However, it has already been demonstrated in different material systems, including also $In_xGa_{1-x}As$ on GaAs.[10–12] As it has been shown, such lowly strained $In_xGa_{1-x}As/GaAs$ QDs have distinguishably different properties than the standard ones, for instance, strong shape asymmetry, low surface density, and large sizes.[12–14] All those properties can be interesting from the application point of view. Strongly asymmetric dashlike structures can be used as an active medium of lasers, where the asymmetric island orientation has to be matched to the laser cavity in order to maximize the optical gain and to minimize the threshold current of the laser.[15] However, low dot densities are usually necessary in all the quantum cryptography and quantum computing applications,[16] whereas large dot volumes inducing enhanced exciton oscillator strength are necessary to increase the strength of light-matter interaction between the QD excitons and photons confined in resonant cavities,[17] which has been already confirmed experimentally for the case of low content and large $In_{0.3}Ga_{0.7}As/GaAs$ dots in pillar microcavities.[14]

Our studies focus on the optical properties of low content and low strain $In_xGa_{1-x}As$ QD structures (i.e., in the composition range of 30% indium and about 2% layer/substrate lattice mismatch), which are barely known yet and for which, in addition, no experimental results on the wetting layer (WL) thickness have been published. In order to investigate their properties we use a combination of scanning electron microscopy (SEM) and optical spectroscopy in a form of photoluminescence (emission spectra) and photomodulated reflectance (absorptionlike spectra). The usefulness of the latter one for studying the QD structures has been demonstrated several times for the case of more common highly strained self-assembled QDs.[18–20] Observations of the

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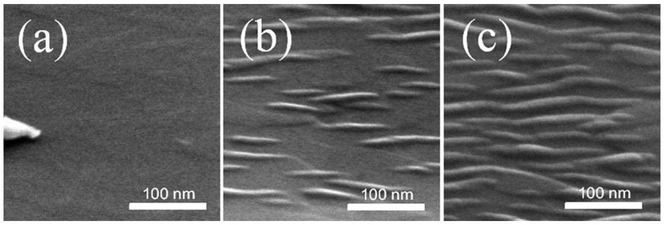

FIG. 1. Surface SEM images for three uncapped structures with In content: (a) 27%, (b) 30%, and (c) 33%.

changes in the optical spectra for various strain structures (from below to significantly above the critical thickness for island formation) have been made and have allowed explaining the origin of the QD layer related features. The absorption character of photoreflectance (PR) and its high sensitivity to optical transitions has finally allowed determining the WL thickness in such a lowly strained $In_{0.3}Ga_{0.7}As/GaAs$ QD structure in a contactless and nondestructive manner and showing how it is altered by small indium content changes.

EXPERIMENTAL DETAILS

Our PR experiment has been realized in the so-called dark configuration of the PR setup.[21] A halogen lamp served as a source of the probe beam light, which then was dispersed by a double-grating 1 m focal-length monochromator. The quasimonochromatic light was reflected off the sample and its intensity was detected by a silicon photodiode. The 632.8 nm laser pump beam was provided by a He–Ne laser for this experiment. This beam was modulated by a mechanical chopper at a frequency of 125 Hz. The samples were mounted on a cold finger in a continuous flow cryostat allowing the measurements at liquid nitrogen or liquid helium temperatures. Photoluminescence (PL) has been measured at standard experimental configuration using an argon laser as an excitation source and liquid nitrogen cooled charge coupled device (CCD) camera as a detector.

The QD structures were grown in an Eiko solid source molecular beam epitaxy (MBE) system. The base surface was an undoped GaAs orientated along [001] direction. All QD structures were fabricated by depositing a 300 nm GaAs buffer layer followed by an $In_xGa_{1-x}As$ QD layer grown via submonolayer deposition.[12] The nominal thickness of this QD layer was only estimated on the base of growth conditions to be about 4.5 nm and was kept constant for all the structures. The growth temperature for the GaAs buffer layer was 590 °C and 510 °C for the low content and low strain dot layers, respectively. For SEM experiments, a set of samples was left uncapped, whereas for optical experiments they were capped with 50 nm of GaAs at the same growth temperature as used for the QD growth. The nominal indium contents have been determined based on the combination of low temperature PL measurements and electron energy loss spectroscopy performed for a calibration set of quantum well (QW) structures, with final accuracy not worse than ±1%.

RESULTS AND DISCUSSION

Figure 1 shows surface SEM images obtained at a 70° tilting angle to enhance the height contrast and taken for three samples grown with a nominally constant InGaAs material amount but differing in indium content from 27% to 33%. The image for the uncapped $In_{0.27}Ga_{0.73}As$ layer [Fig. 1(a)] presents a rather flat and smooth surface (no indications of dot formation) and only some surface roughness due to monolayer fluctuations can be seen. This confirms that the critical thickness had not been exceeded and after capping the InGaAs layer with GaAs, a standard thin quantum well should have been obtained. When the In content is slightly higher (30%) and the critical thickness has been reached the self-assembled growth starts and some islands appear on the InGaAs layer surface [Fig. 1(b)]. Their density is rather low [about $(6-9)\times 10^9$ cm^{-2}] due to a small amount of the material, which could be spent on the dot formation. Finally, for even higher In content (33%), the conditions are definitely above the critical thickness, and this causes the growth of a typical dense QD array (more than 10^{10} cm^{-2}), because even more InGaAs layer material is spent on the dot formation in that case due to a thinner wetting layer thickness for higher In content [Fig. 1(c)]. The typical sizes of these dots are rather large: 20–30 nm in width, 50–100 nm in length, and several nanometers in height, with a tendency for slightly increased size for higher indium contents. As it is seen in Fig. 1, the dots are elongated in shape and preferentially oriented in $[0\bar{1}1]$ direction. The growth and even some possible applications of such large and strongly asymmetric InGaAs/GaAs quantum dots for low In contents have been already reported previously,[10–14] and the reasons for the elongation effect have been discussed.[10–12] However, it should be underlined here that the SEM images in Fig. 1 correspond to the uncapped structures and the shapes and/or sizes of the observed islands might have been modified after the capping procedure.

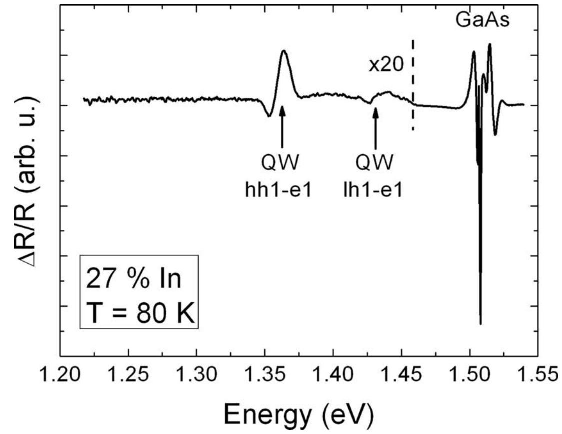

FIG. 2. A PR spectrum for $In_{0.27}Ga_{0.73}As/GaAs$ quantum well.

Figure 2 shows a liquid nitrogen PR spectrum of the sample, which has the lowest In content, i.e., an $In_{0.27}Ga_{0.73}As/GaAs$ quantum well. We could observe two transitions (marked with arrows in Fig. 2) on the low energy side, which we relate to the QW, beside the main one at 1.51 eV related to the GaAs band gap bulklike transition at low temperature. Their energies have been determined basing on the first derivative Gaussian functional form, the most

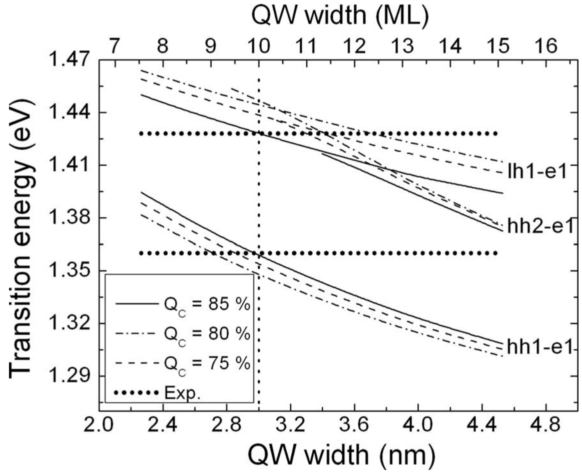

FIG. 3. Energy level calculations for $In_{0.27}Ga_{0.73}As/GaAs$ QW vs well width and for a few band offset ratios (treated as a semi-free parameter).

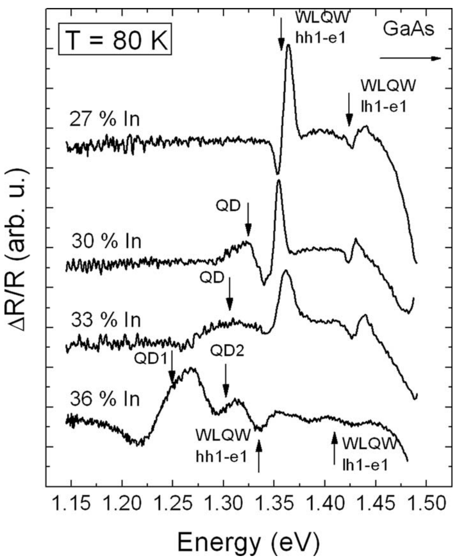

FIG. 4. Low temperature PR spectra for QD structures with various In contents from 27% to 36%.

appropriate PR line shape for inhomogeneously broadened confined state transitions.[21] In order to find out about the origin of the transitions we have calculated the energy levels in a squarelike potential well using simple effective mass approximation in an envelope function approach taking into account the strain effects. The indium content has been assumed to be the nominal one and any effects related to the QW intermixing have been neglected, because they will have a minor influence on the transition energies of a very narrow confinement potential well. Most of the remaining material parameters are well known and have been taken after Ref. 22, a broad review, which gives widely accepted values. However, at least two important ones have to be treated as semi-free due to some uncertainty in their values, namely, quantum well width (which is determined just from the growth conditions) and band gap discontinuities between unstrained well and barrier materials, i.e., the so-called unstrained or theoretical band offset Q_c. The latter one can be limited to quite a narrow range. Usually, the experimentally determined value of the band edge discontinuities has concerned the strained materials or, in other words, the practical band offset (existing in the real structure, which is strain influenced and strain dependent). As it has been shown previously, the strained conduction band offset ratio for $In_xGa_{1-x}As/GaAs$ material system depends rather weakly on the In content for compositions with $x \geqslant 0.1$ and equals to about 0.6 (which is equivalent to $\sim$0.8 of the unstrained one for our composition range).[23,24] On the other hand, it has been demonstrated theoretically and experimentally that the unstrained conduction band offset ratio for pure InAs/GaAs heterointerface falls into the range between 83% and 92%.[24–26] Therefore, we performed our calculations only for several Q_c values around 80%. An example of the calculation result for 27% indium well and a method of the QW width and unstrained band offset determination using the experimental transition energies is shown in Fig. 3. First of all, it is seen that the best agreement has been obtained for the well width of about 3 nm (ten monolayers) and the theoretical conduction band offset ratio of about 85%, which corresponds to 64% of its strained equivalent and is in agreement with previously published data.[23,24,27] Second, that the two transitions we have observed in the PR spectra are the ground state heavy hole and light hole related ones, respectively, and that for this well width and the resulting offsets these are the only possible transitions, i.e., only one confined state exists for each, electrons, heavy holes, and light holes. Some discrepancy between the calculated value of the well width and its technological equivalent can be explained due to limited precision of its calibration during the MBE growth, which was done using very short shutter opening times of about 0.1 s (see the growth description above). For more accurate nominal layer thickness determination, the times needed for opening and closing the shutter, the so-called shutter transients, should be taken into consideration in order to give the effective total time when it was open, which is surely shorter than the nominal one and hence the technological thicknesses are somewhat overestimated.

A comment should also be given regarding the line broadening of the WLQW-related PR features, which equals about 12 meV for the ground state transition at low temperature. This value seems to be larger than might be expected for a quantum well with high quality interfaces. However, one needs to remember that the wells we deal with are very thin ones and therefore even a very small thickness fluctuation will immediately cause a significant spectral broadening of a particular transition (the energy levels will shift significantly when the well width is changed by a monolayer only). As it can be seen in Fig. 3, for the QW thickness range of about 3 nm [10 ML (monolayer)], one monolayer thickness fluctuation corresponds to the ground state transition energy change of more than 10 meV. This fully explains the experimental linewidths, and in spite of their apparent large values this confirms a very high QW fabrication quality (a total thickness fluctuation within a single monolayer).

Figures 4 and 5 show PR and PL spectra for all four investigated samples in the energy range below the GaAs band gap, measured at liquid nitrogen temperature for PR and liquid helium for PL. Comparing them, we can distin-

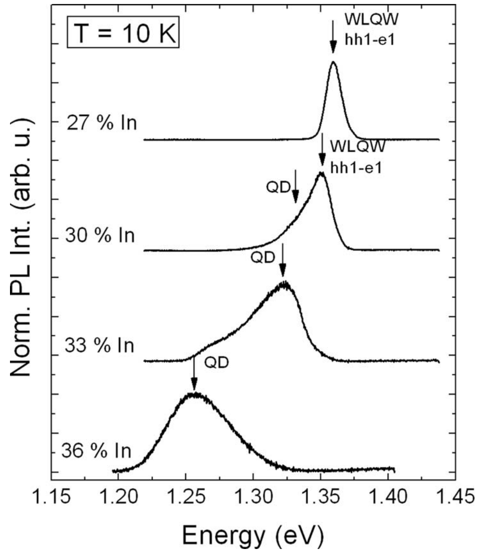

FIG. 5. Low temperature PL spectra for QD structures with various In contents from 27% to 36%.

guish two parts when going from GaAs energy to lower energies, related to quantum well (WL) and QD transitions, respectively. It is seen that for the 27% indium content QW structure a relatively narrow PL peak is observed (13 meV of full width at half maximum, in agreement with PR linewidth) at the energy position of the heavy hole related PR feature, which confirms our previous interpretation. When the In content is increased to about 30%, some QD islands are already created (the critical thickness has been exceeded) according to what is seen in Fig. 1(b). Therefore, we have both WLQW and QDs in the structure. Its PL spectrum shows a double peak, with the main maximum of the narrower line at the energy of about 1.35 eV in agreement with the respective PR feature. It shows the energy of the ground state heavy hole WLQW transition. The light hole related one is seen in the PR spectrum at the energy of about 1.425 eV, and both are marked by the arrows accordingly. The broad low energy shoulder of the PL spectrum can be related to the emission from the dots, whose density is still low enough that, first of all, the radiative recombination via the WL states is still efficient, and second it is not clearly detected as an independent PR line. PR measures normalized changes in the reflectivity coefficient and reflects the absorption of the sample. Therefore, the PR signal intensity is always low for QDs, because they cover only a small fraction of the total illuminated sample surface, and it can be even undetectable for very low dot densities. However, similar as in PL, some low energy wing can be seen on the side of the PR WLQW heavy hole feature, being probably the fingerprint of the measured reflectivity changes (absorption in fact) related to a low density dot array. It becomes already very well pronounced for the structure with 33% indium, where the dot density is significantly higher. There is an inhomogeneously broadened peak in PL (emission through the WL is not effective anymore) and its energy position agrees with the respective PR QD feature. The PR spectra for both 30% and 33% indium structures still include the signal related to the WLQW, allowing the determination of the WL thickness using the procedure shown in Fig. 3. An analogous situation is observed for a QD layer with 36% indium content. Quantum dot emission in PL is in agreement with PR spectrum and WL-related transitions are observable in PR as well.

A comment should be also added regarding the line shape of the PL peak for the 33% indium sample. It might be seen that the spectrum possesses a low energy shoulder or even consists of two peaks in fact, one with the energy closer to the QD peak for 30% content and explained above as the QD related, and second, weaker, with energy close to the QD line of of the 36% sample. We believe that the latter is a signature of a double peaked size distribution of the dot ensemble, i.e., dots with two dominating but significantly different sizes are present in the structure with 33% indium. Similarities in their PL peak energies to lower and higher content structures can be explained as follows. For the case of 30% indium we have a low density array of the smaller dots only. For 33% the dot array not only becomes denser but also some larger islands appear (emitting at lower energy), and we deal with two dot families, and finally for 36% we have a relatively dense array mainly of the larger ones. We do not resolve the double structure for the 33% sample in photoreflectance, which can be understood taking into consideration the absorptionlike character of the PR spectra. Simply, the possible low energy spectral feature (related to a probably very small number of larger dots) gives a negligible contribution, and it is completely hidden by the inhomogeneous broadening. Therefore, the lack of a distinct lower energy feature in PR does not have to be in contradiction to the PL results. Simply, the total absorption connected with these dots remains very small, whereas some important fraction of the total radiative recombination can still go through this channel, because this is the lowest energy state of the system.

The observed QD related PL and PR features shift to the red with the increased In content (strain), which would just suggest that the dots indeed have not only higher planar density but also larger sizes. The second redshift component is the increased indium composition, which, however, could not cause such a strong energy shift only by itself. Additionally, in the PR spectrum of the 36% In content dots a second QD transition has been resolved (labeled by QD2 in Fig. 4). It could be related to the excited state transition in the dots. Simply, when the dots with increasing In content become larger (and the QD material band gap decreases with In content), the 3D confinement potential for carriers becomes effectively deeper, and the confinement energy is larger too (where the barrier height is defined by the WL). Therefore, for smaller composition QDs only one QD confined state line is observed in both PL and PR, whereas for larger dots (36% indium case) we could observe the first excited state transition as well. It is seen in absorptionlike spectrum only (PR), probably because the excitation density used in the PL experiment was too low to fill the higher order states. The energy distance between these two transitions (ground state QD1 and excited state QD2), which is a figure of merit of the confinement strength, has been estimated to be about 60 meV, based on the fitting procedure of the PR spectra [using first derivative Gaussian line shape fit, as the most

TABLE I. Determined WL thickness based on PR spectra for InGaAs/GaAs QD structures with various In contents expressed in nanometers and monolayers.

In content (%)	WL thickness (nm)	WL thickness (ML)
30	2.7	9.0
33	2.3	7.7
36	2.2	7.3

appropriate for the confined states (exciton-like) inhomogeneously broadened transitions]. It is only slightly less than the typical values for smaller and higher-strained $In_{0.6}Ga_{0.4}As/GaAs$ and InAs/GaAs dots[28,29] and comparable to the energy difference for large InAs/GaAs QDs designed to emit at 1.3 μm.[20] The reason is that the confinement in the direction of the largest dimension (of the order of 100 nm) is negligible similarly as for quantum dashes (the state ladder is only quasidiscrete for this direction and energy level distance is surely covered by the inhomogeneous broadening) and its influence could be only detected by single dot spectroscopy. So, the main level discretization is decided by the two remaining sizes, which are not so much different as, for instance, for large InAs/GaAs dots emitting at 1.3 μm.

In general, it could also be possible that the QD2 feature is connected with the existence of the smaller dot population in the 36% structure (if they exist there and if their density is high enough to be probed in PR). The observed QD2–QD1 energy separation is similar to that observed in PL of the 33% structure between the main peak (smaller dots) and the weak low energy one (larger dots). Therefore, none of the two possible origins of the QD2 line can be excluded by our experiments.

We have also determined the QD line broadening from PL and PR spectra in order to estimate the dot homogeneity changes (sizes, shapes, or content distribution) versus their density. The obtained PL and PR linewidths are similar (in the sense of full width at half maximum) and equal to 29, 41, and 54 meV for 30%, 33%, and 36% samples, respectively (i.e., the spectral broadening and, hence, inhomogeneity, tend to increase for higher densities). This also reflects relatively high nonuniformity of the dot properties in comparison to the best (or even typical) values reported for highly strained InGaAs/GaAs and InAs/GaAs self-assembled QD structures.[29] Such a high inhomogeneity makes this kind of dots suitable for broadband emission applications.

Finally, the WL quantum well transitions have been analyzed in a similar way to those observed for 27% QW structure. Again, as it could be expected, the best agreement has been obtained for the unstrained conduction band offset ratio of about 85% (in agreement with the above-presented discussion), and, as a result, the width of the quantum well could be obtained. As these wells are formed in the samples with self-assembled QDs, it means that the values we have determined have the sense of the wetting layer thickness (critical thickness for 2D-3D growth transition) for three various content lowly strained $In_xGa_{1-x}As/GaAs$ QD structures. Their values are summarized in Table I. It is seen that the general tendency is correct: They are larger than those reported for InAs/GaAs or $In_{0.6}Ga_{0.4}As/GaAs$ QDs,[19,30,31] and they decrease with increasing In content, reflecting the higher strain in the structure.

CONCLUSIONS

In conclusion, we have investigated the optical properties of lowly strained InGaAs/GaAs QD samples grown by MBE with indium contents of about 30%, varied in such a way that structures below and above the critical InGaAs layer thickness for 3D island formation have been obtained. We have used PR, a modulation-type spectroscopy, combined with common PL and supported by SEM in order to understand the influence of the processes occurring in both growth regimes and interpret the optical transitions. The absorption character of the modulated reflectivity spectra has allowed us not only to detect the QD response including higher energy transition for the highest content structure (36% of indium) but also to derive the WL thickness versus In content in such a lowly strained $In_xGa_{1-x}As/GaAs$ QD system.

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