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Chinedu Christian Ahia, Ngcali Tile, Amalia Navarro, et al.

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Long wavelength stacking induced shift of the near-infrared photoluminescence from unintentional MOVPE grown InGaSb/GaSb quantum wells

Chinedu Christian Ahia,[1,a] Ngcali Tile,[1] Amalia Navarro,[2]
Beatriz Galiana Blanco,[2] and Johannes Reinhardt Botha[1]
[1]*Department of Physics, Nelson Mandela University, PO Box 77000, Port Elizabeth, South Africa*
[2]*Department of Physics, Universidad Carlos III de Madrid, Spain*

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The effect of stacking on the near-infrared photoluminescence (NIR-PL) of InGaSb/GaSb quantum wells (QWs) which were inadvertently formed during an attempt to fabricate stacked InSb/GaSb quantum dots (QDs) using atmospheric pressure Metalorganic Vapor Phase Epitaxy (MOVPE) are investigated in this work. The morphology of uncapped dots was studied by means of scanning probe microscopy (SPM) which shows a significant deviation in the shape and density of dots grown directly on the buffer compared to those that terminated an "embedded-dot" sample. Cross-sectional scanning transmission electron microscopy (STEM) and transmission electron microscopy (TEM) of the capped structures clearly revealed the formation of QWs in the capped structures. An increase in the number of InSb QD-layers, which metamorphosed into QWs, was observed to cause an increase in the luminescence spectral line width and a long-wavelength shift of the QW PL lines, together with an enhancement in the strength of PL emission. Variations in layer thicknesses and alloy composition introduced as a result of inter-diffusion of Ga and In which is enhanced by the prolonged annealing time of the QDs (during spacer/cap layer deposition) and In adatom migration is suggested to alter the morphology of the capped dots and induce a change in PL peak positions and the spectral linewidth of the NIR low energy lines. © *2018 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/).* https://doi.org/10.1063/1.5037296

INTRODUCTION

The fabrication of low dimensional semiconductor structures has attracted much attention because of their wide spread applications in opto-electronic devices. A reduction in the dimension of semiconducting materials from bulk three-dimensional (3D) semiconductors to zero-dimensional (0D) quantum dots (QDs) gives rise to striking changes in the behaviour of these materials, due to the confinement of electrons and/or holes. QDs function like artificial atoms and have unique characteristics that are significantly different from those of bulk material.[1]

Vertical stacking of QD layers have been reported to induce the nucleation of the individual QDs on top of each other, a characteristic attributed to the strain effect of the underlying QD, provided that the spacer thickness is not too large.[2] Various studies have been performed to examine the influence of stacking on low dimensional systems, such as QDs. Experimental results and theoretical predictions[3] have confirmed that the geometry of dots and their distribution can be enhanced by stacking. This could be achieved through the growth of successive dot layers of specific thickness (separated by a spacer), which invariably stimulates the fabrication of dots with uniform size and spacing, considering

[a]Corresponding author e-mail address: chinedu@aims.ac.za

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the fact that the overlying islands are prone to mirror the buried islands and simultaneously nucleate directly above it. Multilayers of QDs have also been reported[4] to be crucial for effective infrared absorption; however, stacking can also result in the formation of defects due to strain relaxation, while the stacking array of atomic layers during growth process influences the periodicity of the atomic arrangement in the material.[5]

InSb/GaSb QDs are a promising alternative material system for mid-infrared (MIR) applications. Irrespective of the epitaxial technique adopted, the development of InSb/GaSb QDs has been reported[6,7] to be challenging due to the relatively weak indium-antimony (In-Sb) binding energy, which gives rise to a long migration length of In adatoms on an Sb-terminated surface. Many studies[8–10] have reported PL emission of InSb/GaSb QDs within the near-IR (NIR) region; however, transmission electron microscopy (TEM) studies reported recently[11,12] have illustrated the inadvertent formation of an InGaSb quantum well (QW) after capping of InSb QDs, which gave rise to PL spectra similar to what has earlier been reported by some groups[8–10] to be emission from InSb QDs within the NIR region. Emission within the MIR region have been reported[6] for this material system. To date, a consensus is yet to be reached with regards to the emission wavelength of InSb/GaSb QDs. In this paper, we extend our previous investigations[11,12] and report on the effect of stacking on the NIR-PL properties of unintentional InGaSb/GaSb QWs, formed during an attempt to fabricate capped InSb/GaSb QDs.

EXPERIMENTAL DETAILS

A schematic cross-section of the intended structures is shown in Figure 1. The samples were grown on a GaSb ((100) 2° off towards <111>B ± 0.1°) substrate using a Thomas Swan MOVPE reactor, with a horizontal quartz tube operated at atmospheric pressure. The total gas flow was regulated at 2.15 slm, with palladium diffused H_2 as carrier gas, while trimethylindium (TMIn), triethylgallium (TEGa) and trimethylantimony (TMSb) were used as source precursors. A more detailed description of the growth procedure and sample preparation has been reported elsewhere.[11,12] All the layers in the samples used for this study were deposited without growth interruption.

Four samples (referred to as samples 1, 2, 3 and 4) were investigated. The first three samples contained a 300 nm GaSb buffer layer and a 200 nm thick GaSb cap layer, with different numbers

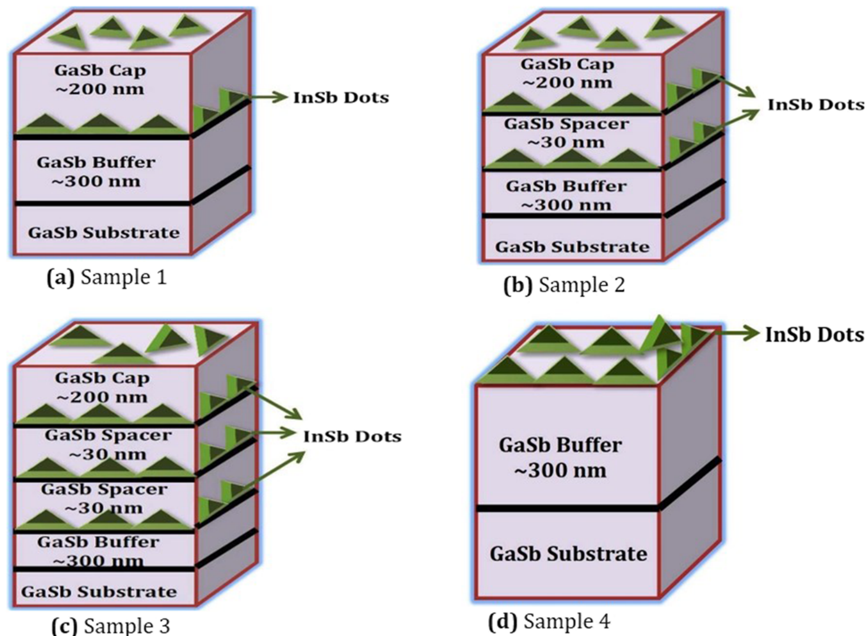

FIG. 1. Cross-sectional representation of intended samples.

of InSb QD layers embedded between the GaSb buffer and cap layers for each sample, while the fourth sample in which uncapped InSb dots were grown directly on a 300 nm GaSb buffer layer was used as a reference for SPM studies. Sample 1 contained a single InSb QD layer, while samples 2 and 3 contained a stack of two and three InSb QD-layers, respectively. Each QD layer in sample 2 and 3 was separated by a 30 nm-thick GaSb spacer layer. For samples 1, 2 and 3, an additional layer of uncapped InSb dots was deposited on top of the final GaSb layer under the same conditions as the embedded InSb dot layers for SPM studies. All four samples were deposited using identical growth conditions. The GaSb cap and spacer layers were grown at a temperature of 500°C at a typical growth rate of ~0.5 Å/s, while the InSb dots were deposited for 5 s at 480°C at a nominal growth rate of ~2.7 Å/s. Prior to the growth of the dots, the 300 nm thick GaSb buffer layer was grown at a temperature of 550°C and a growth rate of ~1.7 Å/s. The V/III ratio was fixed at 2.0 for the growth of the buffer layer and was subsequently increased to 7.0 during the deposition of the spacer and cap layers respectively, while the InSb dots were deposited using a V/III ratio of 1.0. A diode pumped solid state laser operating at 532 nm was used for optical excitation of the samples, which were placed in a closed cycle helium cryostat. The NIR luminescence was detected using a liquid nitrogen cooled germanium photo-detector and analysed using a fully automated Czerny-Tuner type monochromator. The structural features of the capped samples were analysed in bright field (BF) and dark field (DF) imaging conditions, using a double Cs corrected JEOL ARM 200F TEM and a Philips Tecnai 20F FEG TEM, while information on the elemental composition of the samples was deduced using energy dispersive X-ray spectroscopy (EDS) method. Lamellae for electron transparency were prepared using a focused ion beam (FIB), protecting the sample surface through pre-deposition of a platinum (Pt) layer, and milling with Ga+ ions close to normal incidence.

RESULTS AND DISCUSSION

Features such as the dot densities, heights and diameters, were deduced from SPM measurements on the uncapped dots in each sample, using the Bruker NanoScope analysis software (version 1.40). The sample containing no embedded dots (sample 4) had a dot density of the order of $>10^{10}$ cm^{-2}, with lateral dimensions between 30 nm and 80 nm, and height between 4 nm and 12 nm. However, a significant reduction in dot density (~ 8×10^9 cm^{-2}) was noticed for the other three samples (samples 1 to 3, which nominally contained embedded dots). Also, the lateral dimensions increased to between 40 nm and 100 nm. The reduction in uncapped dot density for samples 1 to 3 (shown in the SPM image in Figure 2) is inferred to result from the condensation of Sb on the GaSb capping layer surface at low (non-ideal) temperatures.[13] This can enhance Sb segregation and alter the geometry of the dot.

A comparison of the SPM images displayed in Figure 2 reveals a significant deviation in the shape of dots grown directly on the buffer (see Fig. 2(a)) compared to those that terminated an

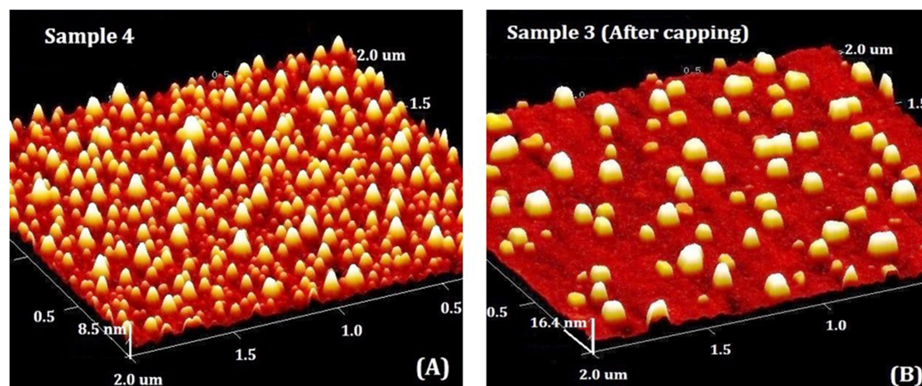

FIG. 2. SPM images showing the morphology and density **(a)** of uncapped dots for sample 4 grown directly on a GaSb buffer, and **(b)** of uncapped dots terminating the cap layer of sample 3.

"embedded-dot" sample, illustrated by the SPM image for sample 3 in Figure 2(b). The former has a pyramidal form with steep side facets, while the latter appear to have a truncated cone shape. The existence of strain at the surface of the samples due to the buried islands and the annealing step of the embedded dots during the deposition of the spacer and cap layers can alter the surface structure of the cap and hence modify the dot morphology. Other factors which can modify the final surface of the cap and consequently, the uncapped dot geometry include the difference between the buffer and cap layer thicknesses, and the variation in their respective growth temperatures from 550°C to 500°C during deposition.

The presence of the capping layer is expected to induce strain in the buried dots besides protecting the QDs from exposure to ambient conditions and is also fundamental in tailoring the band gap of the material. In order to detect luminescence, it was necessary to cap the dots. The TEM/STEM micrographs obtained for samples 1 to 3, shown in Figure 3, reveal the transformation of the intended InSb QDs into QWs after capping. The dissolution of QDs have been reported[14] to occur progressively during the capping process. Factors such as the capping layer material, capping temperature and growth procedure are known to be vital to retain the dots after the deposition of the cap layer. In this study, a high growth rate ∼ >3.5Å/s (which in principle will reduce the time of annealing of the embedded dots and assist in retaining the original morphology) was found to be unfavourable for the deposition of a cap or spacer layer - it led to the propagation of growth defects such as threading dislocations. However, the prolonged annealing of the dots during the growth of the cap or spacer layer(s) at a low growth rate clearly induces intermixing/alloying which destroyed the buried QDs.

According to the STEM and TEM micrographs in Figure 3(a–c), the presence of QWs at the interface between the GaSb cap, spacer and buffer layers suggests the occurrence of inter-diffusion

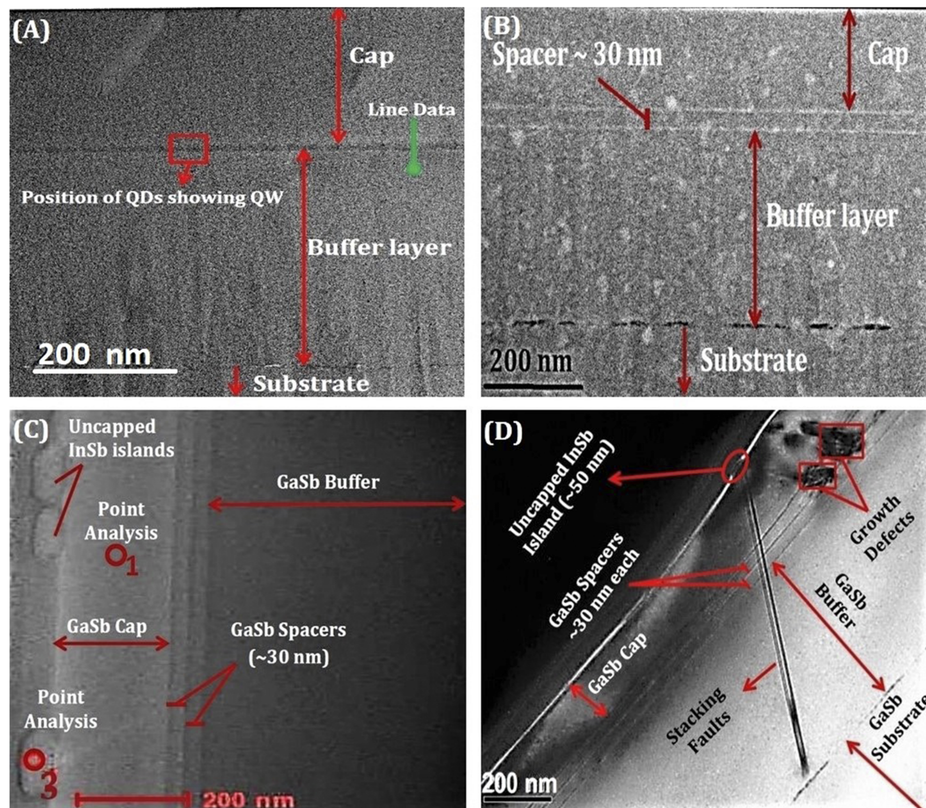

FIG. 3. **(a)** BF cross-sectional TEM micrograph of sample 1, with the green line representing the position where an EDS line scan was performed. **(b)** DF cross-sectional TEM micrograph of sample 2. **(c)** BF cross-sectional STEM micrograph of sample 3, with the red circles ($\mathbf{O_1}$ and $\mathbf{O_3}$) indicating the position where EDS spot analysis was performed, and **(d)** BF cross-sectional TEM micrograph of sample 3 showing stacking faults.

of gallium (Ga) and indium (In) in the region(s) where the buried InSb dots were intended to be. The uncapped InSb islands terminating the samples appear to be unaffected, as seen in Figure 3(c) and (d). Some growth-induced defects were observed in sample 3 at the interface where the dots were expected to be, as shown in Figure 3(d). These defects appear to be introduced as a result of precipitation/formation of Sb crystallites or Ga/In droplets during growth process, which perhaps enhanced the aggregation of islands around this region, thereby promoting defect nucleation and incorporation. According to a previous study,[15] the mutual nucleation of two islands can stimulate the interaction of defects in neighbouring islands, thereby forming extended defects at the boundary.

Other features noticed in sample 3 in Figure 3(d) include a pair of stacking faults generated due to the disruption of the crystallographic planes during growth. The stacking faults appear to originate from the substrate and then extend through the buffer, spacer and cap layers towards an uncapped InSb island (width ~50 nm) on the surface. This is shown more clearly in Figure 4. The bright white line that runs along the surface of the capping layer as seen in Figure 4 originates from the mask (usually platinum) which was deposited over the area of interest and serves as a protective layer for the sample against damages such as scratches during ion milling.

The EDS line-scan shown in Figure 5 was obtained from sample 1 at the position shown in Figure 3(a). The line-scan confirms the presence of In, Sb and Ga at the interface where the dots were expected. Many experimental studies and theoretical reports[16–20] (mostly for InAs/GaAs QD structures) have confirmed that substantial Ga and In inter-diffusion between the wetting layer (WL) and the 3D islands can alter the chemical composition of both of these from that of the actual deposited material.[21]

The STEM micrograph of sample 3 displayed in Figure 6 was taken under high angle annular DF imaging conditions in scanning mode. Significant white contrast is seen in Figure 6. An EDS spot analysis carried out in this region (analysis spot indicated by the red symbol $\mathbf{O_2}$) confirmed the presence of indium in this region. Similarly, the presence of Ga and Sb within the interface region was also established as shown in Figure 7 (labelled spot 2), bearing in mind that only InSb was deposited at the interface region. Galluppi *et al.*[22] were the first to detect In/Ga intermixing in nominally pure InAs dots on GaAs, and noted that the PL spectra from atomic layer molecular beam epitaxy (MBE) grown InAs QDs coincided energetically with those of $In_{0.5}Ga_{0.5}As$ QDs grown using MOVPE. Their results indicated that Ga and In inter-diffusion, in addition to indium segregation, may play a role in QD self-assembly. Acapito *et al.*[21] confirmed this result by using extended X-ray absorption fine structure measurements to determine the composition and the state of strain in $In_xGa_{(1-x)}As/GaAs$ QDs using a first shell bond length analysis. The measurements revealed an alloyed strained WL with

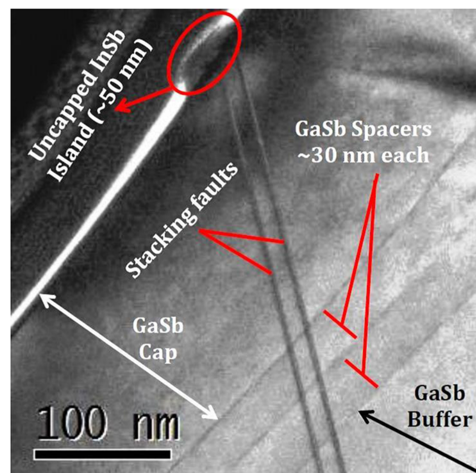

FIG. 4. A magnified image of Figure 3(d) clearly showing the stacking faults terminating at an uncapped dot on the surface of sample 3.

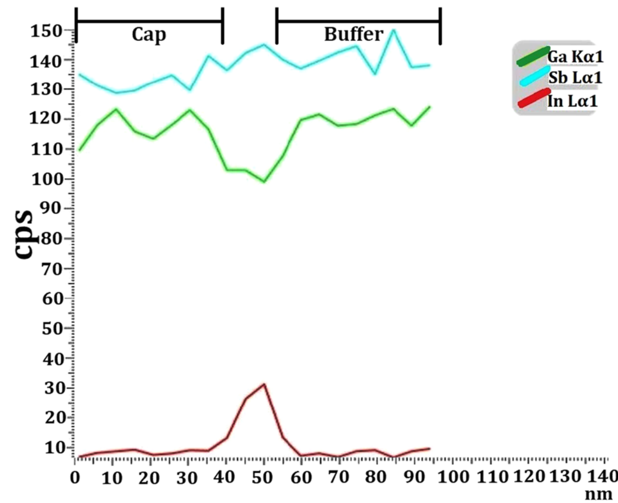

FIG. 5. EDS line-scan taken from sample 1, across the interface between the GaSb cap and buffer layers.

≈15% indium concentration while the QDs comprised of a relaxed $In_xGa_{(1-x)}As$ alloy with an indium content x≈40%. Similarly, by means of TEM and composition profiling, the intermixing of Ga/In in InAs/GaAs self-assembled QDs has been reported[23] to cause a reduction in the maximum indium content in the WL from the actual amount deposited. Factors such as lower growth temperature was described to give rise to slightly weaker intermixing,[23] while the surface migration/diffusion of In atoms out of the top of the QD has also been reported[24] to influence intermixing and the dissolution of the QD apex.

The results of the EDS spot scan displayed in Figure 7 were taken from sample 3, with the analysis spots represented by the red symbols $\mathbf{O_1}$**,** $\mathbf{O_2}$ and $\mathbf{O_3}$ in Figure 3(c) and 6. Results of the EDS scan on the cap layer (labelled spot 1 in Fig. 7) confirmed that the content of the cap layer is Ga and Sb, with no trace of indium. However, the detection of gallium and indium within the interface region where the embedded dots were intended is an indication of inter-diffusion. Penev *et al.*,[25] studied the strain dependence of diffusion and has shown that indium adatom diffusivity on the GaAs (001)-c(4×4) surface can be influenced considerably by the presence of strain. Likewise, results from the experimental confirmation of indium migration processes during the fabrication of InAs/GaAs

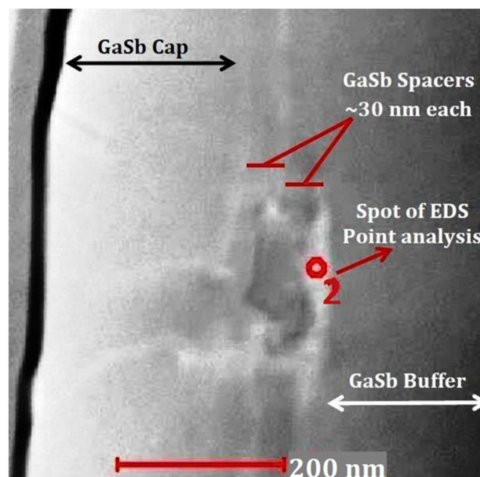

FIG. 6. DF cross-sectional STEM micrograph showing the interface (and area of EDS spot analysis) between the buffer and cap layers where the dots are embedded in sample 3.

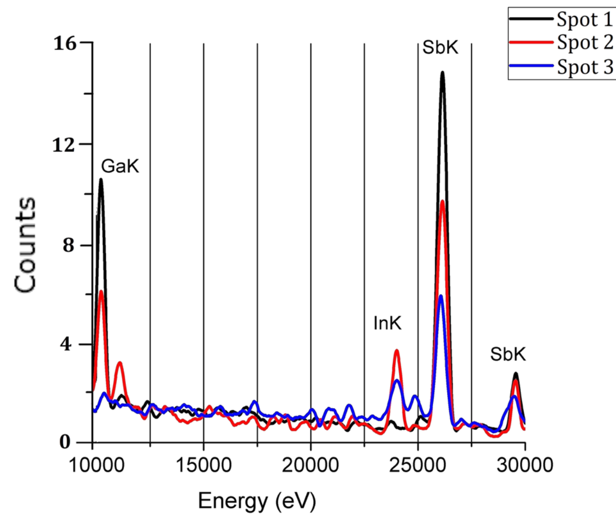

FIG. 7. EDS spot analysis of cap layer region (depicted as spot 1), the interface between the buffer and cap layers where the dots are embedded in sample 3 (depicted as spot 2), and the uncapped dots terminating the cap layer of sample 3 (depicted as spot 3).

quantum posts,[26] has proved that the detachment and surface migration of indium adatoms due to non-uniform distribution of strain can lead to a transient reduction of stress during the deposition of indium.

EDS spot analysis was carried out on an uncapped island terminating sample 3 at the spot indicated by the red symbol $\mathbf{O_3}$ in Figure 3(c), to determine whether or not Ga was present. The results are labelled spot 3 in Figure 7. No evidence of Ga was detected, unlike in the capped islands where significant amounts of Ga were observed. The uncapped islands thus only contain indium and antimony. This supports the suggestion that the capping layer very often causes inter-diffusion, which accounts for the presence of Ga in the intended WL/QD layer, as shown in Figures 5 and 7.

Figure 8 shows the normalised low temperature (10 K) PL spectra obtained from samples 1 to 3, as well as that of the GaSb substrate used. Sample 4, which contained the uncapped dots gave no PL emission, a behaviour which is ascribed to non-radiative recombination at the surface. All the spectra in Figure 8 were collected using an excitation power of 105 mW. Three distinct low energy lines (absent in the substrate material) at ~748 meV, 740 meV and 733 meV can be associated with

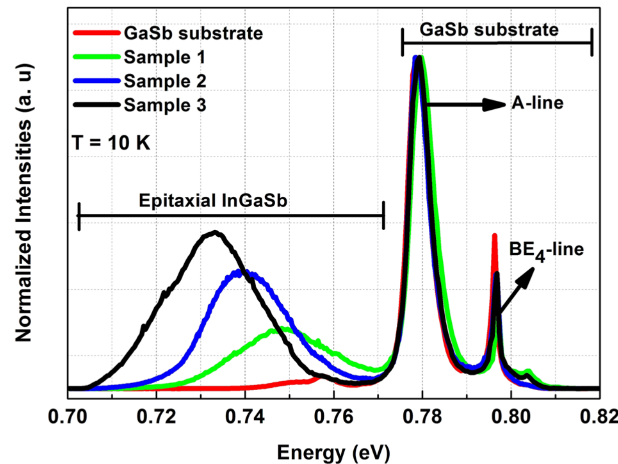

FIG. 8. Normalized low temperature PL spectra of samples 1 to 3. A typical PL spectrum of the GaSb substrate is included for comparison.

the epitaxial material in the grown samples, while the other two PL lines (denoted by A and BE_4[27,28]) have their origin in the substrate. The positions of the low energy lines, believed to originate from unintentionally formed InGaSb/GaSb QWs, vary for the three samples. Sample 3, which contains three QWs, has the strongest low energy emission. The full width at half maximum (FWHM) of the emission lines were measured to be ~18 meV, 20 meV and 25 meV for samples 1, 2 and 3, respectively. Also, the emission wavelength red shifted as the number of layers increases, despite having been deposited under similar growth conditions. A similar red shift of the PL emission line was observed[29] from the PL spectrum of three sheets of vertically coupled InSb QDs, separated by 5 ML GaSb spacers; stacking of the QDs was reported to induce a long wavelength shift of the QD PL lines, due to coupling between the dot layers. A 92 meV red shift in the PL emission line was first reported for multilayer, vertically coupled InAs/GaAs QDs grown by MBE.[30] The red shift was simultaneously accompanied with a 25% reduction in PL linewidth, as the number of InAs islands (~4 nm high) separated by a GaAs spacer layer (~5.6 nm thickness) was increased from a single layer to a stack of 10 layer and was attributed to vertical coupling of islands arranged in columns.[30]

The NIR low energy emission in the present work results from recombination in QWs rather than in QDs. The uncapped dots were transformed due to alloying, resulting from inter-diffusion of Ga and In at the interface where the embedded dots were intended. The extent of inter-diffusion is most likely to be enhanced due to indium adatom migration. Also, as the number of layers in the samples is increased, the effective annealing time of already deposited material will be prolonged, thus enhancing the effect of inter-diffusion and causing an increased red-shift of the QW emission. This may give rise to an inhomogeneous strain distribution, less uniform layers in terms of width and compositions that differ substantially from that of the InSb intended. This can account for the spectral shift in PL peak position and changes in emission linewidth. A detailed description of the low energy PL lines in this study and their behaviour with laser power and temperature has been reported elsewhere.[11,12]

CONCLUSION

The NIR-PL behaviour of stacked InGaSb QWs which were grown during an attempt to fabricate InSb QDs on a GaSb ((100) 2° off towards <111>B ± 0.1°) substrate was presented. The annihilation of the QDs is presumed to occur during the capping process and deposition of the spacer giving rise to QWs. An increase in the number of stacked InSb dots layers which were transformed to QWs was observed to induce a long wavelength shift of the low energy PL lines, and simultaneously prompting an increase in the FWHM and the intensity of the PL spectra from the samples. The long wavelength shift and changes in spectral linewidth of the NIR low energy lines from the epitaxial InGaSb QWs is suggested to be stimulated by variations in layer thicknesses and alloy composition introduced as a result of inter-diffusion of Ga and In which is enhanced by the prolonged effective annealing time of the QDs (during spacer/cap layer deposition) and In adatom migration. Results from this study indicate that inter-diffusion can be promising as a method for controlling the optical properties of QWs and tailoring the spectral response to desired emission wavelengths.

ACKNOWLEDGMENTS

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