InSb quantum dots for the mid-infrared spectral range grown on GaAs substrates using metamorphic InAs buffer layers

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Abstract
Type II InSb/InAs quantum dots (QDs) were successfully grown on GaAs substrates using three different metamorphic buffer layer (MBL) designs. The structural properties of the resulting metamorphic InAs buffer layers were studied and compared using cross-sectional transmission electron microscopy and high resolution x-ray diffraction measurements. Photoluminescence (PL) originating from the InSb QDs was observed from each of the samples and was found to be comparable to the PL of InSb QDs grown onto homo-epitaxially deposited InAs. The 4 K PL intensity and linewidth of InSb QDs grown onto a 3 μm thick InAs buffer layer directly deposited onto GaAs proved to be superior to that from QDs grown onto an InAs MBL using either AlSb or GaSb interlayers. Light-emitting diode structures containing ten layers of InSb QD in the active region were subsequently fabricated and electroluminescence from the QDs was obtained in the mid-infrared spectral range up to 180 K. This is the first step towards obtaining mid-infrared InSb QD light sources on GaAs substrates.

Keywords: quantum dot, mid-infrared, metamorphic growth, interfacial misfit dislocation

(Some figures may appear in colour only in the online journal)

1. Introduction

The mid-infrared spectral range (2–5 μm) is technologically important for many applications including gas sensing because it contains the fingerprint absorption lines of a variety of toxic, pollutant or nuisance gases [1, 2]. In recent years, room temperature mid-infrared lasers have been realized from type-I quantum wells [3], inter-subband quantum cascade and type-II interband cascade structures [4, 5]. However, many of these designs are complex to fabricate and semiconductor quantum dots (QDs) could provide an interesting simpler alternative approach towards producing mid-infrared lasers, which may result in low threshold current density, reduced Auger recombination and high efficiency due to the delta-shaped density of states in QDs [6]. Previously, we have successfully grown type II InSb/InAs QDs by using molecular beam epitaxy (MBE) [7] and liquid phase epitaxy [8], and obtained photoluminescence (PL) between 3 and 4 μm up to room temperature [9]. In addition, we have demonstrated room temperature InSb QD light-emitting diodes (LEDs) grown onto InAs substrates [10]. Integrating InSb QDs onto GaAs substrates would provide access to the mid-infrared spectral range, using a more convenient and inexpensive substrate with associated mature processing technology, opening up new possibilities for device applications. However, because InSb QDs need to be grown within an InAs matrix, to deposit these QDs on GaAs substrates one must first accommodate the large lattice mismatch between InAs and GaAs (Δa/a0 = 7.2%). Depending on the growth conditions, during the initial stages of InAs deposition on GaAs either self-assembled islands or 2D films can be formed [11, 12]. Experimental work has shown that during MBE growth the strain becomes fully relaxed after 7 nm of InAs has been deposited under indium rich conditions [13]. Consequently, a high density of threading dislocations can be expected to appear in the epilayer, which serves as the major strain relief mechanism [14]. Previous studies of...
thick InAs epilayers on GaAs substrates have revealed that the structural and transport properties of InAs are dependent on the substrate temperature during the MBE growth [15]. However, thick buffer layers are often undesirable and besides direct metamorphic growth, other techniques can also be used for the growth of highly lattice-mismatched materials by MBE. For example, the epitaxial growth of GaSb on GaAs substrates has been demonstrated by inserting AlSb interlayers with different thickness and surface treatments [16], such that a 1.2 nm thick AlSb interlayer can give the smallest roughness and best interface quality [17]. Another approach is to intentionally form an interfacial misfit (IMF) dislocation array with large lattice mismatch, in which the strain energy is released through the IMF technique, followed by a 0.5 μm thick InAs buffer layer using an Sb-to-As anion exchange reaction [20]. For all the present samples, 0.8 monolayer of InSb was deposited during the growth of each QD layer. The InAs spacer between QD layers was set to be 20 nm and the samples were each capped with a 200 nm layer of InAs. The samples were grown on semi-insulating GaAs (0 0 1) substrates using a VG-V80H MBE reactor. In situ reflection high energy electron diffraction was used to monitor surface reconstruction. In each case, at the beginning of the growth, a 15 nm thick GaAs layer was first deposited to smooth the substrate surface. The InAs buffer layers were all grown at 550 °C. The AlSb interlayer in sample (B) and the GaSb interlayers in sample (C) were deposited at 620 °C and 600 °C, respectively. The substrate temperature was reduced to 490 °C for the growth of QDs layers and the InAs spacers in all samples.

In this work, we investigated and compared the use of different buffer layer designs to accommodate the lattice mismatch between InAs and GaAs and then evaluated the properties of the InSb QDs grown on the resulting InAs metamorphic buffer layer in each case. High resolution x-ray diffraction (XRD) and cross-sectional transmission electron microscopy (TEM) techniques were used to study the strain, interfaces and the dislocations in all the grown samples. The optical properties of both the buffer layers and the QDs were examined using PL spectroscopy and were compared with QDs grown onto homo-epitaxial InAs, (lattice-matched on InAs substrates). Subsequently, electroluminescence (EL) from the InSb QDs on GaAs was examined from p–i–n diodes fabricated containing ten layers of InSb QDs in the active region.

2. Experimental procedures

As shown in figure 1, three different metamorphic buffer layer designs (MBLs) were used for the growth of InAs on GaAs substrates before the start of the InSb QD growth. In sample (A), a 3 μm thick InAs buffer layer was grown directly on GaAs. In sample (B), an AlSb interlayer was first deposited on the GaAs, followed by a 0.5 μm thick InAs buffer layer. In sample (C), a thin GaSb layer was grown on the GaAs substrate using the IMF technique, followed by a 0.5 μm thick InAs buffer layer. In all three samples, ten layers of InSb QDs were grown on top of the respective InAs buffer layers using the same growth parameters. The InSb QD growth originates from an Sb-to-As anion exchange reaction [20]. For all the present samples, 0.8 monolayer of InSb was deposited during the growth of each QD layer. The InAs spacer between QD layers was set to be 20 nm and the samples were each capped with a 200 nm layer of InAs. The samples were grown on semi-insulating GaAs (0 0 1) substrates using a VG-V80H MBE reactor. In situ reflection high energy electron diffraction was used to monitor surface reconstruction. In each case, at the beginning of the growth, a 15 nm thick GaAs layer was first deposited to smooth the substrate surface. The InAs buffer layers were all grown at 550 °C. The AlSb interlayer in sample (B) and the GaSb interlayers in sample (C) were deposited at 620 °C and 600 °C, respectively. The substrate temperature was reduced to 490 °C for the growth of QDs layers and the InAs spacers in all samples.

Cross section TEM specimens were prepared using conventional methods and examined in a JEOL 2000 FX operating at 20 kV. The structural quality of the resulting epilayers was examined using high resolution double crystal XRD measurements using a Bede QC200 diffractometer. PL measurements were performed on all samples at low temperatures using an Oxford Instruments variable temperature continuous flow helium cryostat. An Ar+ ion laser (514 nm wavelength) was used for excitation of the sample with a maximum incident power density of 10 W cm⁻² at the sample. The emitted mid-infrared radiation was collected using CaF₂ lenses and focused into a 0.3 m Bentham M300 grating monochromator. The signal was detected using a liquid nitrogen cooled InSb photodiode detector and a Stanford Research (SR850) digital lock-in amplifier. Edge emitting p–i–n diodes with a broad area (75 μm wide) ridge structure were subsequently fabricated using conventional photolithography and two step wet etching (1:1:1 H₃PO₄:H₂O₂:H₂O followed by 1:8:20 H₃SO₄:H₂O₂:H₃O). Electrical contacts were made by evaporating Ti/Au (20 nm/200 nm) on both sides of the devices. The diode chips were mounted and wire-bonded onto To46 headers for testing. EL was excited using 20 kHz current.
pulses of up to 1.0 A at 1% duty cycle supplied from an Agilent 8114A pulse generator and measured using the same experimental setup as used for the PL measurements.

3. Results and discussion

3.1. TEM observations

Cross-sectional TEM images were taken for all the three samples in order to investigate the interface structure and dislocation density. Figure 2(a) shows the cross-sectional TEM image of sample (A), where the inset shows details of the InSb QD region. As expected, a large density of threading dislocations is nucleated at the interface between the GaAs and the InAs buffer layer, after which the threading dislocation density becomes reduced with increasing InAs thickness. It is clear from the TEM picture that, after 3 μm thick InAs growth, the dislocation density becomes much lower than at the interface. Although QD layers can be used for dislocation filtering [21], in our case threading dislocations were seen to penetrate through several layers of QDs, as shown in the inset of figure 2(a), which is perhaps because of the sub-monolayer thickness of our InSb QDs. In fact it may be noticed that the first few QD layers actually result in the formation of some additional dislocations. For the exchange growth of InSb QDs, the indium cell temperature needs to be reduced in order to lower the indium flux. During the growth of the first few QD layers, the temperature may not have fully stabilized. The spacing between the adjacent QD layers at the bottom is a little larger compared with the spacing between the top QD layers, consistent with a higher indium cell temperature which results in a higher InAs deposition rate.

Figures 2(b) and (c) show the cross-section TEM images of sample (B) and sample (C), respectively, with the insets giving details of the interlayers. From figure 2(b), the IMF periods are visible at the AlSb/GaAs interface, with a measured period of about 5.7 nm. A smooth surface can be formed at the top of the AlSb interlayer and there is no large density of dislocations observed within the AlSb region from the TEM image. Under these growth conditions, self-organized trenches with non-uniform spacing were automatically formed in the AlSb layer, with a 55° {1 1 1} side wall angle to the substrate surface, as specified by the red arrows in figure 2(b). In sample (C), the IMF features at the GaSb/GaAs interface with a measured period of about 5.9 nm can also be observed. The IMF periods in both samples are very close to those reported earlier [22, 23]. Unlike sample (B), a continuous GaSb layer can be formed on GaAs and the density of dislocations can be effectively reduced within the GaSb layer. However, when depositing the InAs buffer on top of either AlSb or GaSb, new dislocations are once again generated, as shown in figures 2(b) and (c). The TEM images also indicate that the dislocation densities both near the interface and in the QD region are very similar between samples (B) and (C). From the TEM images we cannot reliably determine which interlayer is more helpful in suppressing the threading dislocations. However, when comparing images in figures 2(a)–(c), the lowest density of dislocations in the QD region still occurs in sample (A). In a very similar manner to sample (A), the variation of the QD layer spacing and additional dislocations arising from the bottom QD layers was also observed in samples (B) and (C).
Figure 3. (a) $\omega$–2$\theta$ XRD spectrum of sample (A), the expected periodic side peaks from the QD layers are illustrated by the black curve. (b) $\omega$–2$\theta$ XRD spectrum of sample (B), the stronger tail on the left-hand side shoulder of the InAs peak is from the discontinued AlSb interlayer. (c) $\omega$–2$\theta$ XRD spectrum of sample (C), the weaker peak on the left-hand side shoulder of the InAs peak is from the GaSb interlayer. (d) $\omega$–2$\theta$ XRD spectrum of sample (D), the side peaks are fitted by a MQW model (black curve). (e) The $\omega$-scans of the InAs peak in samples (A), (B) and (C).

3.2. X-ray diffraction

Figures 3(a)–(c) show the $\omega$–2$\theta$ XRD scan of samples (A), (B) and (C), with the XRD spectrum of a reference sample of ten layers of QDs grown on InAs substrate (sample (D)), as shown in figure 3(d). Two major peaks, one from the GaAs substrate and the other from the InAs MBL layer, both exist in all samples. Although the InAs buffer layers have different thicknesses, the position of the InAs peak is almost the same in each case, which confirms that the strain originating at the interface is almost 100% relaxed in all the samples. The $\omega$-scans of the InAs peaks from samples (A), (B) and (C) are also given in figure 3(e). Because of the thick InAs layer in sample (A), the intensity of the InAs peak in XRD is about five times stronger than from the other two samples. The measured FWHM of the InAs peak in sample (A) (236 arcsec), is also significantly smaller than that in sample (B) and sample (C) (483 arcsec and 416 arcsec, respectively). This indicates that the InAs buffer layer in sample (A) has achieved smaller crystalline mosaicity than the other two samples [24], where the higher density of threading dislocations can affect the InAs layer growth more seriously. We can also notice weak fluctuations on both sides of the InAs peak in sample (A), which probably originate from the QD layers. In a similar manner to multiple quantum wells [25], the multiple QD layers in our samples would be expected to give periodic peaks on both sides of the InAs peak, as the XRD spectrum of sample (D) shows. However, due to the changing thickness of InAs spacer layers between the InSb QD layers, such periodic features cannot be clearly observed from sample (A), and are absent from samples (B) and (C). In sample (C), a clear peak appears on the left-hand side of the InAs peak which we attribute to the GaSb interlayer. In sample (B) the AlSb interlayer only results in a wide tail, which is probably because of its discontinuous nature.

3.3. Photoluminescence

The PL from the InSb QDs grown on top of the InAs MBL can help give an indication about the quality of the surrounding
InAs. Previous work has shown that the density of InSb QDs grown using the exchange technique on InAs results in a high QD density of $\sim 10^{12}$ cm$^{-2}$ [7]. PL spectra measured from the three samples at 4 K using an excitation laser power of 10 W cm$^{-2}$ are shown in figure 4, together with the PL spectra from sample (D), which is from the InSb QDs grown on homoepitaxial InAs under optimized QD growth conditions. In all the measured samples, both the dominant peaks from the InSb QDs and the emission from bulk InAs can be observed. The much higher relative PL intensity from the QDs than from the InAs in each of the three samples is an indication of successful growth of high density QDs using all the three metamorphic techniques. The inset of figure 4 shows the magnified PL spectra between 2.9 and 3.3 $\mu$m of the samples when excited with a lower laser power (1 W cm$^{-2}$) at 4 K, in which different recombination mechanisms in InAs become more clearly revealed. Besides the peak at around 2.98 $\mu$m (peak 1) from band-to-band transitions, there is a peak at 3.1 $\mu$m (peak 2) in all samples except sample (C), which originates from bound exciton transitions and has been previously observed in the low temperature PL of InAs epilayers grown on GaAs [26, 27]. According to [27], the absence of bound exciton transitions can be correlated with poor luminescence efficiency of the InAs epilayer, which in our case indicates the InAs layer in sample (C) is not of equally good quality as in samples (A) and (B). In addition, another peak near 3.25 $\mu$m (peak 3) can be observed in the spectrum of sample (A) only. Its transition energy (382 meV) agrees well with the donor–acceptor pair (DAP) recombination in InAs grown on GaAs [26, 27]. It is still possible that DAP recombination is also taking place in other samples, but it cannot be clearly resolved since it lies very close to the much stronger QDs emission in these samples.

Comparing the InSb QDs PL spectra between samples (A) and (D), we find that both PL peaks are approximately Gaussian, originating from the size distribution of the QDs. The QDs in sample (A) also achieved similar PL peak intensity to that of the QDs grown on homoepitaxial InAs (sample (D)). The change in PL peak position between these two samples (from 3.56 $\mu$m to 3.36 $\mu$m, respectively) results from differences in growth conditions which affect the dimensions and size distribution of the resulting QDs [28]. The linewidth of the QD PL peak in sample (A) is 270 nm which is only slightly larger than that of 223 nm in sample (D). These observations indicate that, (i) the density and size distribution of QDs is very similar in samples (A) and (D), (ii) although the density of dislocations in the vicinity of the QDs in sample (A) as seen from TEM images remains high, it has no significant effect on the PL of the QDs. This is probably not surprising since the QD density is a few orders of magnitude higher than the dislocation density. As shown in figure 4 the QDs PL spectra from samples (B) and (C) both have significantly stronger tails on the longer wavelength side and the peak positions have shifted to around 3.36 $\mu$m, even though the QDs were grown under nominally the same conditions as sample (A). As demonstrated in [28], the growth conditions strongly determine the resulting QD size, shape and density. The much thicker InAs buffer layer in sample (A) can still make a difference to the heat transfer on the sample surface when the QDs were being grown, making the actual surface temperature slightly different for sample (A) compared with the other two samples during the QD growth. This can result in a significant change of PL peak position and intensity, which explains the differences in peak wavelength between sample (A) and samples (B) and (C). The PL peak intensity from sample (B) is still higher than sample (C), and the PL spectrum tail is weaker. We can suppose that...
Figure 5. Integrated QD PL intensities from samples (A), (B) and (C) at different temperatures. The dotted lines show the exponential fit of PL intensity with temperatures below 60 K, and the dashed lines show the exponential fit for temperatures above 60 K. The inset shows the relation between the logarithm of integrated PL intensity from QDs ($\ln(I)$) and the reciprocal temperature ($1/T$) from which the activation energy can be extracted. The straight lines give the linear fit in the low $1/T$ region.

A higher density of QDs with better size uniformity has been grown in sample (B) than in sample (C), which also indicates the AlSb interlayer is more helpful than the GaSb interlayer for the subsequent growth of good quality InAs. However, the PL intensities from these two samples are still weaker than in sample (A). We believe that is because the top of the 0.5 μm thick InAs buffer above the (AlSb or GaSb) interlayer growth cannot yet serve as an equally good pseudo-substrate for growth of high quality InSb QDs under the same growth conditions.

The QDs PL from samples (A), (B) and (C) can be measured up to 180 K, 170 K and 140 K, respectively. The integrated PL intensities at different temperatures are plotted in figure 5 which shows that for all the samples investigated, the PL quenching with increasing temperature accelerates significantly above 60 K consistent with thermal escape of confined holes from the QDs [29, 30]. The thermal activation energy can be calculated from an Arrhenius plot, as shown in the inset of figure 5. The activation energies of 80, 64 and 62 meV for samples (A), (B) and (C) are approximately in agreement with the heavy hole confinement energies obtained from $k\cdot p$ modeling [31]. The higher activation energy in sample (A) corresponds to QDs having deeper hole confinement, which is consistent with a longer wavelength PL peak emission associated with formation of larger QDs. The difference in corresponding transition energies for (B) and (C) is also consistent with their reduced thermal activation energies. Details are given in table 1.

### 3.4. Electroluminescence

The highest PL intensity was obtained from QDs with a thick InAs buffer (sample (A)) and so as shown in figure 6, edge-emitting p-i-n LEDs containing ten layers of QDs in the (i-) active region were grown by MBE on a p-doped ($\sim 2 \times 10^{18} \text{ cm}^{-3}$) GaAs substrate with the direct metamorphic deposition of InAs (as in (A) above). The intrinsic region was composed of a 4 μm thick InAs layer containing the ten sub-monolayer insertions of InSb QD layers in the middle, each with a 20 nm spacing. The upper layer consisted of a 2 μm thick n-doped ($\sim 2 \times 10^{18} \text{ cm}^{-3}$) InAs layer. EL emission was extracted from the 75 μm wide edge of the 1 mm long ridge structure using 1 A current pulsed excitation (20 kHz, 1% duty cycle). The measured spectra from 10 up to 180 K are shown in figure 6, with the inset illustrating the diode structure. The highest peak at 3.07 μm at 10 K originates from recombination in the InAs, while the broader emission at longer wavelengths is from the InSb QDs. Contrary to the PL results, the EL from the InAs layer is always stronger than the EL from QDs. During the EL measurement, higher densities of electron–hole pairs are injected to the sample than in the PL measurement so that an overflow of carriers into the InAs can occur, resulting in more overall emission from the InAs. The temperature dependence of integrated EL intensities for both the InAs and the InSb QDs is illustrated in figure 7. The
EL of the InSb QD is insensitive to temperature below ~60 K and quenches more slowly than the PL of the InAs, which is dominated by non-radiative Auger recombination. In EL, the injected carrier density is much higher than in PL and holes are constantly being provided into the QDs by the applied electric field. This reduces the net rate of escape of holes out of the QDs and helps to maintain the EL emission intensity. Consequently, the decay of the InSb QD EL emission is less rapid with temperature than in PL. These results are in good agreement with our earlier findings from InSb QD LEDs grown on InAs substrates which exhibited EL at room temperature with the addition of an AlₓGa₁₋ₓAs₀.₅Sb₀.₅S electron blocking layer [10]. The EL from the QDs in the present structures does not persist up to room temperature partly for this reason.

4. Conclusions

In order to incorporate InSb quantum dots (QDs) on GaAs substrates, we have experimented with the direct metamorphic growth of a thick InAs buffer layer, and the insertion of AlSb or GaSb interlayers with thinner InAs buffer layers, to accommodate the strain caused by the large lattice mismatch. In all samples, the QDs were grown on top of the resulting InAs buffer layers. Cross-section transmission electron microscopy (TEM) images showed that the density of threading dislocations in the QDs region grown on a thick InAs buffer is lower than in the other two samples grown using interlayer techniques. The TEM images revealed that interfacial misfit arrays are formed at both AlSb/GaAs and GaSb/GaAs interfaces. In addition, self-assembled trenches can be formed in the AlSb layer.

The 4 K PL intensity and linewidth from InSb QDs grown with direct metamorphic deposition of a thick InAs buffer layer, and the insertion of AlSb or GaSb interlayers with thinner InAs buffer layers, are very close to that of InSb QDs grown homoepitaxially on InAs substrate, even though the threading dislocation density is still high. This is because the QD density is a few orders of magnitude higher than the dislocation density. The top surface of the thick InAs buffer is of a sufficiently good quality to serve as a pseudo-substrate for the QDs growth, and the carrier recombination within the QDs is not sensitive to the existence of a high density of threading dislocations due to the carrier confinement. However, as temperature increases and holes become thermally excited out of the InSb QDs, the quality of the surrounding InAs becomes more significant since non-radiative Shockley–Read–Hall recombination influences the re-capture rate of holes. Consequently, further optimization of the metamorphic buffer layer would be beneficial in achieving room temperature mid-infrared electroluminescence (EL) from InSb QDs in InAs grown on GaAs substrates.

The x-ray diffraction measurements confirmed that the thick InAs buffer has better crystallinity than the InAs grown on interlayers. In order to reduce the thickness of the InAs buffer layer, the use of an AlSb interlayer appears to be a better choice than the GaSb interlayer. Finally, prototype p–i–n diodes containing InSb QDs were made using direct growth of a thick InAs buffer, and mid-infrared EL at 3.6 μm was obtained from InSb QDs in non-optimized edge emitting light-emitting diode structures up to 180 K.

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Table 1. Summary of the QD PL emission results and activation energies from samples (A), (B) and (C).

| Samples | PL peak energy (meV) | Wavelength (μm) | FWHM (nm) | Activation energy (meV) | Heavy hole confinement energy (meV) |
|---------|---------------------|----------------|----------|-----------------------|----------------------------------|
| A       | 349                 | 3.56           | 270      | 80                    | 71                               |
| B       | 369                 | 3.36           | 255      | 64                    | 49                               |
| C       | 369                 | 3.36           | 305      | 62                    | 47                               |

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