N-incorporation and photoluminescence in In-rich InGaAsN grown on InAs by liquid phase epitaxy

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Abstract:

Dilute nitride InGaAsN layers with high In content have been grown on InAs substrates by liquid phase epitaxy using GaN as a precursor for N in the growth solution. Photoluminescence (PL) was obtained in the mid-infrared spectral range at temperatures between 4-300 K. Although Ga increases the InAs bandgap, the strong band anti-crossing effect from the N incorporation resulted in an overall bandgap reduction of 11 meV compared with InAs. The temperature-dependent PL exhibited a complicated behavior and showed an anomalous increase in intensity from 190 K to room temperature. This was due to the formation of a complex defect which behaves as a non-radiative recombination centre and prevents radiative band-band recombination at temperatures < 190 K. Above this temperature the PL increases as band-band transitions become allowed. The formation of this defect requires the presence of both Ga and N and becomes de-activated after a high temperature anneal. Raman spectroscopy confirmed the presence of phonon modes associated with In-N and Ga-N bonds confirming the incorporation of N using liquid phase growth.

1. Introduction

The dilute III-V-nitride semiconductors are attractive for fundamental research as well as for a variety of practical applications [1-4]. Because of the availability of cheaper substrates and mature GaAs technology, a large amount of work has been reported on GaAsN and quaternary InGaAsN layers grown on GaAs where the In content is less than 10%. Addition of In to GaAsN compensates for the strain produced by nitrogen and lattice matched layers have been grown on GaAs for In concentrations of 7% and nitrogen content of 2% for applications including high efficiency multilayer solar cells[5]. With 3 at% nitrogen, the material has been used for the fabrication of diode lasers emitting in the 1.3-1.55 μm optical communication range [6-8]. There are also a few reports on `high indium` InGaAsN with an In content $> 50\%$ and which is grown lattice-matched to InP substrates [9-13]. From theory, it has been established that nitrogen-induced band gap reduction in these materials occurs in a similar way to that in low indium content InGaAsN [14] and it exhibits some interesting phenomena, such as, weaker interaction between the nitrogen level and the conduction band as compared to that in low In content InGaAsN [10] and a change in nitrogen bonding configuration upon annealing [12]. Furthermore, for this alloy there are many applications within the technologically important mid-infrared (2-5 μm) spectral range [15, 16] including; laser based remote sensing of pollutant and greenhouse gases, thermal imaging, biomedical analysis and infrared countermeasures for security, as well as in thermo-photovoltaic devices [17]. Although mid-infrared LEDs, lasers and photodetectors have been demonstrated [18-21], the fabrication of efficient and cost-effective light sources and detectors is challenging because of the unfavorable imbalance in the density of states (DOS) due to the light electron effective mass in InAs [22, 23]. Recently, we have shown that it is possible to produce InAsN by molecular beam epitaxy (MBE) with good structural quality which exhibits photoluminescence (PL) emission in the mid-infrared [24, 25]. Provided that growth conditions are optimized, the electrical properties show that the mobility and residual carrier concentration are not so strongly affected by the introduction of N compared with GaAsN [26-28]. Good quality material has been demonstrated both on InAs and on GaAs substrates [29, 30] where the incorporation of N and the material quantum efficiency can be improved by the addition of Sb during growth [31, 32]. More recently, dilute nitride quantum wells which exhibit mid-infrared PL emission have also been demonstrated [33].

The dilute nitrides offer excellent potential to readily access longer wavelengths and many possibilities to engineer novel device structures and to date MBE growth has been used to effectively obtain N incorporation. In this work we report on growth from solution using liquid phase epitaxy (LPE), which may offer an alternative route towards the production of high quality dilute nitrides provided that the N can be successfully incorporated. Previously, we have reported LPE growth of InAsN layers on InAs substrates where polycrystalline InN powder was added to the growth melt to supply N during growth

[34]. In the present work we report on our attempts to improve on this by using GaN as a source of N instead of InN. One advantage of using GaN is that it is now available with 99.999% purity whereas the maximum purity available for commercial polycrystalline InN is 99.98%. We observed that it is possible to use GaN to successfully introduce N into the InAs crystal using LPE. We report on the growth of In-rich InGaAsN layers on InAs substrates with indium content > 97% which exhibit PL in the mid-infrared spectral range and which show some interesting temperature dependence. Raman spectroscopy and thermal annealing was used to probe the local arrangement of atoms around the incorporated N in the crystal.

2. Experimental Procedures

The InGaAsN epitaxial layers were grown using the conventional LPE technique in a horizontal sliding boat LPE reactor. High purity In (6Ns), obtained from *JMC Ltd*, UK, was first baked at 800°C for 20 hrs under Pd-diffused hydrogen flow. The In solution was then saturated with As by placing it over an undoped single crystal InAs wafer at 600°C for 1 hr followed by further baking at 700°C for 15 hr. Next, precisely weighted quantities (upto 2 mol% in the melt) of 5N purity polycrystalline GaN grains (*Kyma Technologies*, USA) were added to the melt and baked at 700°C for 1 hr. Finally, an n⁺ (100) InAs substrate was mounted in the boat and growth was carried out for 5-10 min under a supercooling of 4-5°C and a constant cooling rate of 0.3°C/min. The resulting epitaxial layers exhibited smooth and shiny surfaces with a thickness in the range 3-5µm. The surface texture was found to degrade for GaN content above 4 mol% in the melt.

The Ga content in the layers was measured by energy dispersive X-ray analysis (EDX) using a *Sirion 200* electron microscope. The change in lattice constant of the layer, as a result of both Ga and N incorporation, was observed using high resolution double crystal x-ray diffraction (XRD) measurements. Photoluminescence (PL) measurements were done in the range 4-300K, where the test sample was held in a continuous flow He cryostat. An Ar⁺ ion laser was used for excitation and the luminescence from the sample was analyzed through a 0.3m *Bentham M300* monochromator and detected with a liquid nitrogen cooled InSb detector and a lock-in amplifier. The N-related local vibrational modes (LVM) were characterized using a *Renishaw InVia* micro-Raman spectrometer with a 576 x 400 pixel CCD detector cooled to -70 °C. Excitation was provided by a diode laser (100 mW at 785 nm, which is 1.58 eV, higher than the band-gap of all the InGaAsN samples). A spot size of ~1.6 µm² was obtained using the 50X objective lens of a *Leica DM2500M* compound microscope. The spectral resolution was ~1 cm⁻¹ using a 1200 line/mm diffraction grating and a 50 µm entrance slit. A Rayleigh edge holographic notch filter was

used to block out unwanted reflections. An exposure length of 10 seconds was used to minimize potential heating effects at room temperature, with scans from 100 to 1000 cm^{-1} taken at several locations on each sample. Each scan had 10 accumulations to reduce the effects of noise.

3. Results and discussion

From EDX measurements, we found that the material contained 2.3 at% Ga for growth from a melt containing 4 mol% GaN, which appears to be the upper limit for successful epitaxy of smooth layers. High resolution x ray diffraction spectra for three samples grown with successively increasing amounts of up to 2 mol% GaN in the melt are shown in Fig.1. In all three cases, we observed two clearly distinguishable peaks, one due to the InAs substrate and the other due to the epitaxial layer. Furthermore, with increasing GaN content in the melt, the separation between the peaks increased indicating a decrease of the lattice constant of the layer. Unfortunately, both N and Ga effect a reduction in the lattice constant and hence it cannot be said specifically which species is primarily responsible for the observed decrease in the lattice constant since N is not readily detected using EDX.

Figure 2 shows the 4 K PL spectrum of InGaAsN grown with 0.7 mole% GaN added to the melt together with that from an un-doped InAs homoepitaxial layer also grown by LPE for comparison. The InGaAsN spectrum consists of three peaks located at 2.97, 3.22 and 3.51 μm which have been labeled A, B and C respectively. The un-doped InAs exhibits two characteristic peaks located at 3.01 and 3.12 μm , which correspond to band-band and donor-acceptor transitions respectively, while the smaller peak located at 3.37 μm is attributed to defects or impurities within the band gap [35]. From the comparison, peak A can be attributed to band-band transitions in the InAs substrate, whereas the main peak B may be associated with emission from localized impurity or native lattice defect states close to the InGaAsN band edge. Peak C originates from recombination involving a defect level lying deeper within the InGaAsN band gap.

Figure 3 shows the temperature dependence of the PL from the InGaAsN epitaxial layer. As the temperature increases peak A becomes more prominent while peak B quenches rapidly, consistent with thermal redistribution of carriers from localized states into the conduction band. However, we note that the InGaAsN band-band photoluminescence is not observed until higher temperature (> 160 K) because of strong non-radiative recombination as will be discussed below. Similarly, peak C becomes fully quenched at temperatures above 60 K. Both the peaks B and C exhibit almost no shift in peak position with increasing temperature which is typical of emission from a defect level.

Between 80 K and 190 K, peak A quenches. As the temperature increases above 190 K a new feature appears, beginning at approximately 3.25 μm and which evolves into a new emission band (labeled as D) which then dominates the spectrum. This band can be identified with band–band transitions in the InGaAsN since its behavior follows the Varshni empirical formula quite closely at elevated temperatures, as shown in Fig.4a. The corresponding band gap of the InGaAsN alloy at each temperature was determined by extrapolation from a linear fit to the square of the near-band gap portion of the photoluminescence intensity and is shown in Fig.4b. This is consistent with electron-hole recombination across a direct gap semiconductor with a parabolic density of states (DOS) where, $I \propto (h\nu - Eg)^{1/2}$. The extrapolation of the Varshni fit in Fig.4a results in a value for the InGaAsN band gap as 0.405 eV at 0 K, which is smaller than the generally accepted value for InAs (0.416 eV) and confirms the incorporation of N which reduces the alloy band gap due to the band anti-crossing effect [36]. This is sufficient to counteract any band gap increase arising from the incorporation of Ga. EDX analysis on this sample was unable to detect N directly, but revealed that the Ga content in the InGaAsN was 2%. Consequently, using this value together with the band gap obtained from PL we may estimate the N content to be 0.3 % since the band gap reduction is entirely due to the incorporation of N.

A rather unusual feature of the emission band D is that it grows steadily in intensity with increasing temperature (> 160 K) and continues to increase up to room temperature where the experiment was stopped. This behavior is shown in figure 5 and was observed in all the InGaAsN samples characterized.

In general, PL intensity decreases with increasing temperature because of an enhancement of competing non-radiative recombination which is of the form:

$$I = \frac{I_0}{1 + C \exp\left(-\frac{E_0}{kT}\right)} \quad \dots\dots\dots (1)$$

In our samples we observe a clear deviation from this relationship as is evident from Fig.5. We attribute the anomalous increase in band D to the existence of a non-radiative deep level defect within the InGaAsN band gap which acts as an efficient centre for non-radiative recombination at temperatures below 160 K. Regarding the nature of such a defect, it must be noted that the increased PL at higher temperatures is only observed in materials containing both Ga and N in the epitaxial layer. Figure 6 shows the high temperature PL of an InGaAs epilayer grown with 4 at% Ga in the melt under similar conditions. This material does not demonstrate any increase in emission with increasing temperature and follows closely the relationship described in equation (1). Growth of InAsN by LPE (or MBE) [34,25] also results

in no increase in PL emission. Consequently, the introduction of both Ga and N into the epilayer are likely to result in the formation of a complex deep level defect which changes its ionization state with increasing temperature, switching from an efficient non-radiative recombination centre at low temperature to an inactive neutral centre at higher temperatures which then enables band-band recombination to occur more efficiently. A similar increase in band-band PL with temperature has been observed in GaAsN, although this was interpreted on the basis of a spin blockade involving Ga self-interstitials and relies on preventing the capture of spin polarized free electrons by non-radiative recombination centers with the same spin orientation as the localized electrons [37]. The formation of such features have also been found to be thermodynamically favorable in the presence of N, while no such behavior was found in N-free GaAs epilayers. In our case, since anti-site defects (such as As vacancies and In_{As}) are known to form in InAs, the addition of Ga and N may easily result in the formation of complex defects with multiple charge states [38].

To further elucidate the nature of the defects responsible for the anomalous PL behavior, the sample was subjected to a thermal annealing treatment. The InGaAsN epitaxial layer was placed face down on a clean undoped InAs wafer and annealed at 750 °C for 15 mins in an ultra-pure hydrogen atmosphere. The resulting PL emission is shown in Fig.7. Note that the overall intensity of the PL increased by approximately one order of magnitude (Fig. 5) and the peak at 2.97 μm is greatly increased while that of the peak near 3.22 μm has been drastically reduced. The changes in the PL emission spectrum are most likely due to a re-arrangement of the local site symmetry around the incorporated N and Ga atoms, or the out diffusion of N as a direct result of the post growth anneal [39]. It should also be noted that the higher temperature increase in PL intensity is no longer observed after the annealing, this can also be seen clearly in Fig.5 (solid points). From the Arrhenius plot of integrated PL intensity in Fig.8 we found that the increase of PL intensity from 190 to 300 K in the as-grown material is associated with an activation energy of 26 meV. The activation energies for thermal quenching for the as-grown sample was 9 meV, which changed to 15 meV after annealing.

Fig.9 shows the Raman spectra from two InGaAsN epitaxial layers grown from a melt containing 0.7 and 2 mol% GaN along with the spectrum from undoped InAs for comparison. Strong InAs transverse optical (TO) phonon and longitudinal optical (LO) phonon modes can be readily identified at 217 and 238 cm^{-1} , respectively [40] in all the three samples. A weak band between 250 and 300 cm^{-1} is found in InGaAsN only and can be attributed to GaAs TO (268 cm^{-1}) and LO (293 cm^{-1}) phonon modes [41, 42]. A broad feature extending between 420 and 500 cm^{-1} is again found only in the InGaAsN samples. These peaks arise from the presence of N complexes and N-related local vibrational modes (LVM) in the material [41,

42]. In the InGaAsN quaternary system, the Ga-N bond length (1.95 Å) is less than the In-N bond length (2.14 Å), resulting in a larger bond strength for Ga-N bonds, making their formation more favourable compared to that of In-N bonds. On the other hand, for the same reason, a Ga-N bond in an InAs lattice, with In-As bond length of 2.61 Å, is more strained compared to an In-N bond in the same lattice. According to Klar et al [43], in as-grown InGaAsN, a nitrogen atom surrounded by four nearest neighbor Ga atoms (Ga_4N), is favoured in the surface region, whereas in the bulk, the In_4N configuration is preferred. They further suggested that after a high temperature anneal, the entire material is converted to the latter configuration. From the spectra in figure 8 two prominent LVMs can be identified at ~450 and 470 cm^{-1} associated with configurations such as In_4N [41, 44] and Ga_4N [45], both of these are likely to exist in as-grown InGaAsN. However, it should be noted that it is difficult to unambiguously identify these N-related peaks due to the presence of 2nd order InAs transitions present within the same region [46]. Furthermore, it has been shown experimentally [12] as well as theoretically [47] that even in high In-content InGaAsN containing a small amount of Ga, there is a preferential formation of Ga-N bonds. This explains the almost equal Raman signals due to InN-like and GaN-like LVMs obtained in our samples containing about 3 at% Ga.

The Raman difference spectra between the 0.7 mol% and 0 mol% samples is shown in figure 10 and depicts the region of the broad feature centered around 450 cm^{-1} . From this spectrum 6 peaks are evident located at 380, 413, 439, 461, 487 and 496 cm^{-1} respectively. These peaks are related to the presence of N since the InAs related peaks have been subtracted, (the InAs peaks would normally appear in the minima of this spectrum). The InN-like mode at 439 cm^{-1} can be identified as the $^{14}\text{N}_{\text{As}}$ local vibrational mode consistent with previously published data [12,46]. The two peaks located at 461 and 496 cm^{-1} we attribute to N- In_4 and N- Ga_2In_2 clusters respectively [48]. The peaks at 413 and 487 cm^{-1} are related to higher order complexes and have been determined to be due to high order di-nitrogen In-N complexes [34] and doubly degenerate N- Ga_3In cluster modes [48, 49]. Finally, the peak located at 380 cm^{-1} is tentatively attributed to a gap mode caused by In vacancies [50].

Figures 5 and 8 show that after a high temperature anneal, the PL dependence on temperature reverts to the more usual thermally activated quenching of equation (1). This is consistent with the Raman spectrum of Fig. 11, which shows that the N-related modes observed in the InGaAsN layer grown with 2 mol% GaN in the melt become broadened and shift to higher frequencies near 500 cm^{-1} . This is in agreement with previous findings where such behavior was related to a change in the atomic arrangement around the nitrogen atoms [51, 52]. Although further work is required to fully identify the nature of the clusters and defects present in our InGaAsN it is apparent that using GaN as a source of N results in the formation of

complex defects or clusters involving N which are responsible for the temperature dependent behavior of the PL.

5. Conclusion

In summary, InGaAsN layers with high In content have been grown on InAs substrates using liquid phase epitaxy by adding GaN to the InAs growth melt. Using this technique a maximum incorporation of 2.3 at% of Ga was obtained in the as-grown epitaxial layers. PL was obtained in the mid-infrared spectral range at temperatures between 4-300 K. Interpretation of the temperature dependent PL enabled us to extract the 0 K bandgap as 0.405 eV. Together with the x-ray diffraction and electron microprobe analysis results we were able to obtain the alloy composition as InGaAsN. Although Ga increases the InAs bandgap, the strong band anti-crossing effect from the N incorporation resulted in an overall bandgap reduction of 11 meV. The temperature-dependent PL exhibited a complicated behavior with increasing temperature and showed an anomalous increase in intensity from 190 K to room temperature. From our results, it seems likely that this is due to the formation of a complex defect which behaves as a non-radiative recombination centre. This prevents radiative band-band recombination at temperatures < 190 K. Above this temperature the PL increases presumably as the charge state of the defect changes and band-band transitions become allowed. The defect also becomes de-activated after a high temperature anneal. It further appears that the formation of this defect requires the presence of both Ga and N, as we have not found any such behavior in the PL of ternary InGaAs or InAsN epilayers. Raman spectroscopy confirmed the presence of phonon modes associated with In-N and Ga-N bonds. However, further work is required to elucidate the actual composition, site symmetry and charge states of the N related structure defects present in this material.

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Figure Captions

Fig 1. HRXRD rocking curves for three InGaAsN layers grown with increasing amounts of GaN added to the growth melt

Fig 2. Comparison of the PL at 4 K from undoped homoepitaxial LPE-grown InAs (red) and as-grown InGaAsN with 0.7 mol% GaN (black) added to the In-rich LPE growth solution

Fig 3. PL for as-grown InGaAsN in the temperature range (a) 4-60K and (b) 80-300K. The principal transitions are labeled and an increase in PL emission intensity is evident in (b) as the temperature approaches room temperature

Fig 4. (a) Temperature dependence of peak B (black squares) and emission band D (red circles), the solid red line is a Varshni fit to the data, and (b) the intensity squared vs energy plots used to estimate the InGaAsN band gap from band D at high temperatures by extrapolation

Fig 5. Integrated PL intensity as a function of temperature for as-grown (open circles) and annealed (solid circles) InGaAsN

Fig.6. Near room temperature PL of LPE-grown high indium content InGaAs, showing the usual decreasing intensity with temperature

Fig. 7. PL for annealed LPE InGaAsN in the temperature range (a) 4-120K and (b) 120–300K

Fig.8. Arrhenius plots of the integrated PL intensity for the as-grown (open circles) and annealed (solid points) InGaAsN.

Fig. 9. Raman spectra for two InGaAsN samples, grown with 0.7 and 2 mol% GaN in the melt, compared with that obtained from an undoped InAs sample.

Figure 10: Raman difference spectra of 0.7 mol% InGaAsN . The position of N related configurations is indicated by the arrows above the spectrum while the InAs 2nd order locations are indicated by the arrows below the spectrum.

Fig.11. Raman spectrum for an InGaAsN layer grown from a melt containing 2 mol% GaN, compared with that from the same sample after a high temperature anneal.

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