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Influence of Sb, Bi, Tl, and B on the incorporation of N in GaAs

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GaAs:N is an interesting material for many devices due to its unique compositional variation of band gap. Small amounts of N lead to a strong decrease in band gap energy as well as lattice constant. The further addition of In or Sb leads to quaternary alloys with band gap energies below 1.4 eV lattice matched to GaAs. One drawback of these alloys is the low solubility of N in GaAs. Some success has been obtained using low growth temperatures and V/III ratios during organometallic vapor phase epitaxy to kinetically limit phase separation. This article describes mechanisms for N incorporation into the GaAs crystal during growth and shows how surfactants like Sb, Bi, and Tl, as well as B, affect N incorporation. A decrease of the N content in GaAs was found for Sb, Bi, and Tl, which can be explained using a simple Langmuir model with competitive adsorption. The surface morphology of the epitaxial layers and the influence of surfactants was analyzed using atomic force microscopy. © *2002 American Institute of Physics.*
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INTRODUCTION

Materials with band gap energies of less than 1.4 eV grown lattice matched on GaAs are of great interest for devices such as multijunction solar cells,[1,2] heterojunction-bipolar transistors, and laser diodes.[3,4] Small amounts of N have been found to reduce the band gap energy of GaAs[5] and the GaAs:N alloys can be made lattice matched to GaAs by the further addition of larger atoms such as In or Sb.[6]

Several groups have been working on this promising material with some initial success. One problem is the low equilibrium solubility of N in GaAs. In fact, the calculated thermodynamic solubility is only on the order of 10^{14} atoms per cm^3.[7] Still crystals with up to 5% N in GaAs have been grown by organometallic vapor phase epitaxy (OMVPE),[8] molecular beam epitaxy (MBE),[9] metalorganic-MBE (MOMBE),[10] and chemical beam epitaxy (CBE).[11] Low growth temperatures below 600 °C and small V/III ratios were used in the case of OMVPE growth to kinetically limit phase separation. Under these conditions kinetic effects at the growth surface play a dominant role.

The purpose of this article is to explain some of the factors affecting N incorporation under these growth conditions. For nominally (001) oriented GaAs substrates, the influence of $[\bar{1}10](A)$ and $[110]$ (B) steps was investigated by using intentionally misoriented substrates. Atomic force microscopy (AFM) was used to determine the influence of N on the surface morphology and step structure. Another important question was related to the influence of surfactants like Sb, Bi, or Tl on the N content of GaAs:N. Surfactants are typically elements with small incorporation and desorption coefficients which accumulate on the surface during growth, thus changing its properties. These surfactants can potentially change the adatom sticking coefficients, surface diffusion coefficients, step structure, and surface reconstruction. This has been intensively studied in the GaInP material system where CuPt ordering can in fact be reduced by adding small amounts of TESb, TMBi, or DETe to the gas phase during growth.[12–14]

Recently, it has also been shown that Sb has some influence on the dopant incorporation in GaAs.[15] Small amounts of TESb increase Zn and In incorporation by as much as a factor of 1.6. This effect was attributed to an increase in the surface diffusion coefficients, enabling more Zn or In atoms to reach a step and become incorporated into the solid before desorption.

Other groups have reported an influence of In or Sb on the N incorporation in GaAs:N grown by OMVPE,[16] MBE,[9] and CBE.[11] For OMVPE grown samples, a superlinear dropoff of the N incorporation was found with increasing In content in $Ga_{1-x}In_xNAs$ alloys. In fact, the N content approaches zero at $x=0.5$. Several explanations have been proposed. Indium surface seggregation might lead to an In-rich surface layer, lowering N incorporation.[16] The desorption rate of volatile nitrogen species on the growth surface might also be enhanced by adding indium.[11] Another explanation from Zhang and Zunger is related to the influence of the surface reconstruction on dopant incorporation in III–V semiconductors.[17] Furthermore, gas phase reactions between DMHy and TMIn might lead to the observed effect. However, Friedman *et al.*[16] showed that the DMHy flow has no influence on the In content in $Ga_{0.5}In_{0.5}As$ which makes this argument unlikely. The higher bonding energy between N and Ga, as compared to N and In, was also mentioned as a

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possible cause by Miyamoto *et al.*[11] However, as the incorporation of N in GaAs cannot be explained thermodynamically, the validity of this argument is uncertain.

The observed correlation between N and In content in GaInNAs is strongly dependent on the growth technique and precursor used. Results from MBE and CBE grown samples suggest that In has no significant influence and that Sb actually increases N incorporation in GaAs by 60% for an Sb content of 0.35 in GaAsSbN.[9] This behavior was explained by a higher sticking coefficient of N in the presence of Sb on the growth surface. The group-III flux on the other hand does not seem to have an influence on the N incorporation. Contrary results have been observed in the present work for Sb in OMVPE grown samples. A major difference between OMVPE and MBE or CBE growth is the nature of the nitrogen source. For OMVPE, DMHy is the common source. A radio frequency plasma cell is used for MBE and CBE. This results in N ions or radicals having a small desorption rate upon reaching the growth surface.[11] DMHy on the other hand does not decompose completely at low growth temperatures and therefore, N–C and N–H complexes may dominate the epitaxial surface. These complexes may have lower sticking coefficients resulting in an increased N incorporation with increasing growth rate.[11] The opposite behavior was found for CBE grown layers.

In this article secondary ion mass spectroscopy (SIMS) was used to study the effects of Sb, Tl, Bi, and B on the N incorporation in GaAs. Each atom, except for B, was found to reduce the N content in GaAs grown by OMVPE. For the group-V atoms Sb and Bi this behavior can be explained by a Langmuir model where the reduced N incorporation results from a competition of N and the surfactant atom for the same surface sites. From this point of view one can also understand why a group-III element like B does not have any effect on N incorporation as N and B atoms reside on different lattice sites.

It is unclear why the group-III atom Tl produces a decrease in the N incorporation into the solid. One possible explanation is that the bulky atom Tl energetically affects the neighboring group-V sites. Tl might, therefore, change the sticking coefficient of N complexes on the surface. A second possibility is that Tl competes for the occupancy of the normally group-V sites on the surface. In either of these two cases, one might expect the competitive Langmuir model to describe the results. On the other hand, the Tl precursor might react with the DMHy in the gas phase to reduce the amount of N available for adsorption on the surface. However, this seems unlikely in light of the high ratio of N to Tl in the vapor. One would also not expect this process to be described by the competitive adsorption model.

EXPERIMENTAL PROCEDURE

GaAs:N crystals with surfactants Sb, Tl, Bi, and B have been grown by OMVPE in a horizontal, infrared-heated, atmospheric pressure reactor.[18] Trimethylgallium (TMGa), triethylantimony (TESb), cyclopentadienylthallium $(C_5H_5)_2Tl$, trimethylbismuth (TMBi), 1,1-dimethylhydrazine (DMHy), diborane, and 100% arsine were used as the source materials.

(100) GaAs substrates with both singular and vicinal (3°, 6°, and 15° toward $\langle 111 \rangle A$ and 3°, 6° toward $\langle 111 \rangle B$) orientations have been used. Preparation of the wafers consisted of standard degreasing followed by a 1 min etch in a 2:1:12 solution of NH_4OH, H_2O_2, and H_2O. The substrates were then rinsed in de-ionized water for 5 min and blown dry with N_2 before loading into the reactor.

The carrier gas was Pd-diffused hydrogen. The growth temperature and V/III ratio were 560 °C and 30, leading to a N concentration of from 0.3% to 3% in the GaAs:N alloys. The growth rate was approximately 1 μm/h. The crystals were analyzed using x-ray diffraction (XRD) and low-temperature photoluminescence (PL). The N content was calculated from the XRD peak separation for coherently strained layers[19] and the PL peak separation[5] accounting for a temperature of 12 K.

The 12 K PL was excited using the 488 nm line of an Ar^+ laser with a power of 10 mW focused onto a 0.5 mm^2 spot. The emission was dispersed using a Spex model 1870 monochromator and detected with a silicon diode. This detector was only able to measure GaAs:N samples with N contents below 1.5%. The characterization of the surface structure was carried out using a Nanoscope III AFM in the tapping mode. Etched single-crystalline Si tips were used with an end radius of about 5 nm and a side wall angle of about 35°. An area of 3×3 μm^2 was scanned. The samples were measured in air and therefore are covered with a thin, conformal, native oxide layer.

SIMS depth profiles were measured on GaAs:N multilayer structures to probe the influence of Sb, Tl, Bi, and B on N incorporation. Typical layer structures consisted of up to six layers each with a thickness of 400 nm. The measurements were performed by Applied Microanalysis Laboratory using a Cameca ims-3f system and either a Cs^+ or O^- primary ion beam.

RESULTS

GaAs:N layers have been grown on (100) GaAs substrates with several misorientations in the $\langle 111 \rangle A$ and B directions to investigate the influence of the step density and orientation on the N incorporation. The N content of 31 GaAs:N samples was calculated from the Cu $K\alpha_1$ peak separation of the substrate and GaAs:N layer. Figure 1 shows the difference in the N content of the layers, measured relative to the N concentration of GaAs:N grown on singular substrates versus the substrate misorientation angle.

[110] steps on substrates which are tilted in the $\langle 111 \rangle B$ direction seem not to influence the N incorporation at all. A different behavior was found for $[\bar{1}10]$ steps on surfaces tilted toward $\langle 111 \rangle A$. For small misorientation angles of up to 3°, no influence was found, but an average of 60% less N was measured for a misorientation angle of 6°. This behavior can be explained by the number of bonds that a N atom can form at a step. The typical situation is illustrated in Fig. 2. A N atom attached to a $[\bar{1}10]$ step can only form a single bond to the underlying Ga atom, whereas a N atom at a [110] step forms two bonds, the same number as on a (001) terrace.

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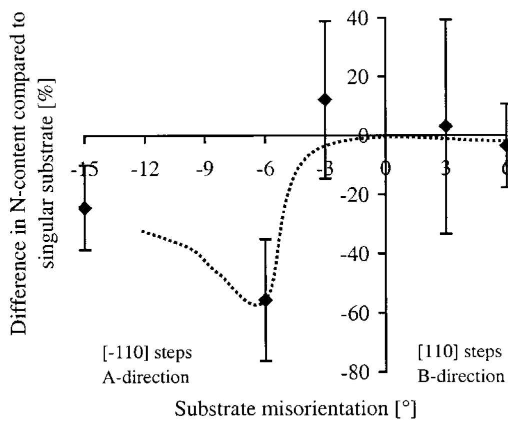

FIG. 1. Dependence of the N content in GaAs:N on the misorientation of the GaAs substrate from (001). The data plotted are the N content of the misoriented sample minus the value of N concentration on singular GaAs. Error bars show the standard deviation for 31 different samples.

Thus, the attachment of N atoms is unfavorable for $[\bar{1}10]$ steps, leading to a reduction of the N content on substrates with a large misorientation toward $\langle 111 \rangle A$.

The surface morphology of GaAs and GaAs:N layers for similar growth conditions of 560 °C, V/III = 30, and a growth rate of 1 μm/h have been investigated by AFM. Figure 3 shows 3×3 μm^2 images of the surface with a depth resolution of 5 nm. These two samples show the influence of N on the step structure and growth mode of GaAs:N with an N content of about 3%. A typical "wedding-cake" morphology with large islands and a constant step spacing was found for both samples. The addition of N to the growth surface seems not to influence the morphology, although it may cause the island shape to be more elongated along the [110] direction.

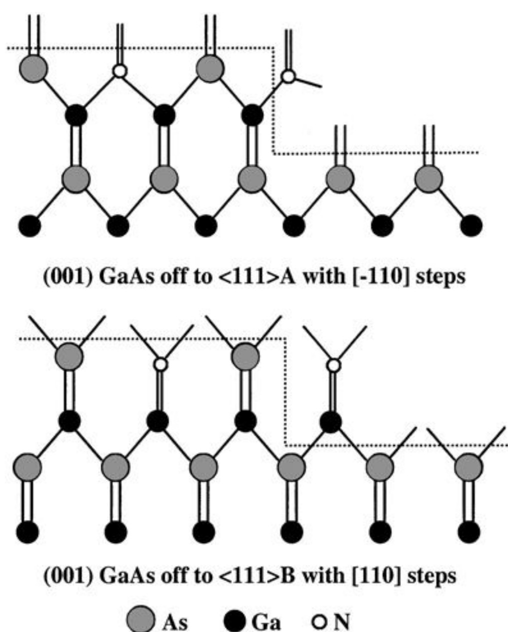

FIG. 2. Schematic cross section of a step on an (001) GaAs surface with a misorientation toward $\langle 111 \rangle A$ (top) and $\langle 111 \rangle B$ (bottom) to produce $[\bar{1}10]$ and [110] steps, respectively.

For the GaAs and GaAs:N samples in Fig. 3, both [110] and $[\bar{1}10]$ steps are found to have similar densities. This might explain why small misorientations in the $\langle 111 \rangle A$ direction do not influence N incorporation significantly. At higher tilting angles the density of $[\bar{1}10]$ steps will increase and finally dominate the adatom attachment leading to a reduction of the N content in GaAs:N. Therefore, the behavior observed in Fig. 1 can be simply explained by taking into account the bonding situation for group-V atoms at [110] and $[\bar{1}10]$ oriented steps and the island growth mode found for these low growth temperatures of 560 °C.

Clearly, the surface step structure affects N incorporation in GaAs. This suggests that other surface effects, such as the addition of surfactants, might also influence the incorporation of N in GaAs. Thus, the effects of the surfactants Sb, Bi, and Tl as well as the influence of B on N incorporation were investigated. Figure 3 shows AFM images of GaAs:N layers grown with small concentrations of $(C_5H_5)_2Tl$, TESb, and B_2H_6 in the gas phase. One can see that TESb has only a minor influence on the morphology, but that both $(C_5H_5)_2Tl$ and B_2H_6 cause a significant roughening of the surface. The rms roughness for samples grown with Tl and B increases to about 2–5 nm as compared to 0.5 nm for GaAs:N grown without the presence of these elements. This behavior is not due to a change in the lattice constant caused by the incorporation of Tl or B into the crystal. The concentrations of both elements in the GaAs:N layers were found to be well below 1%. Therefore, a change in the adatom attachment has to be the reason for this result.

Figure 4 shows the influence of TESb, TMBi, $(C_5H_5)Tl$, and B_2H_6 partial pressure on N incorporation into GaAs:N, grown under otherwise identical conditions. The data are normalized to the N content of GaAs:N grown without surfactant addition. Tl, Bi, and Sb were all found to decrease the incorporation of N in GaAs. B has no observable influence for B_2H_6 concentrations giving approximately 10^{20}B atoms/cm^{-3}in the solid. For higher B_2H_6 partial pressures, the morphology becomes rough and SIMS analysis indicates that the layers are coated by an amorphous BN layer.

Figure 4 also shows data from SIMS analysis, as well as from 12 K PL measurements. The PL samples were double layer structures with and without the surfactant Sb or Tl. The difference in N content was calculated from the energy separation between the peaks with and without surfactant. The error bars represent uncertainties due to the peak width and the variation of results from different samples. Figure 5 shows a typical 12 K PL spectrum of such a sample before and after removal of the top layer by etching. One can see that the low energy peak at 1393 meV disappeared after etching the sample in $NH_4OH:H_2O_2:H_2O = 2:1:12$ for about 1 min. Therefore, the influence of TESb on N incorporation could be clearly identified.

DISCUSSION

Sb, Bi, and Tl have all been found to significantly reduce N incorporation in GaAs grown by OMVPE. The group-V atoms Bi and Sb are expected to compete directly with N for

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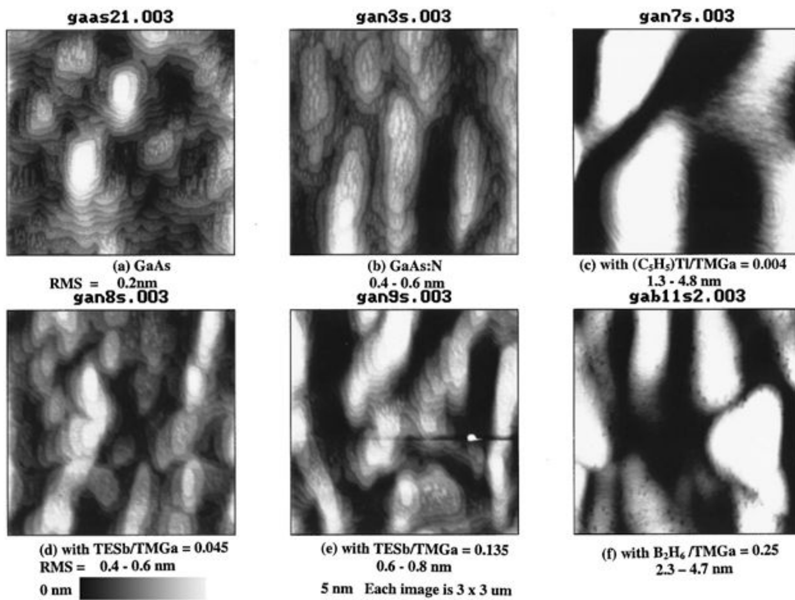

FIG. 3. AFM images of GaAs (a) and GaAs:N surfaces grown under similar conditions (560 °C, V/III = 30, 1 μm/h) with the addition of no surfactant (b), and the surfactants Tl (c), Sb (d), (e), and B(f).

surface sites. Furthermore, these surfactants accumulate on the surface during growth. The atomic concentrations in the solid have been found to be small, $<10^{20}$ cm^{-3} for Sb and $<2\times10^{16}$ for Bi. Nevertheless, the surface concentrations are expected to be high. In this case, the surfactant atoms might effectively block N adsorption and, thus, incorporation of N into the solid.

Such a competitive adsorption of atoms on the surface can be analyzed using the simple Langmuir model.[20] This model is based on the assumption that the surface is composed of a fixed number of sites on which group-V species can adsorb, a fraction (θ_i), of which are occupied. Atoms sitting on the group-V lattice sites compete against each other. In this model, every lattice site on the surface is treated as being equivalent and interactions between species are ignored. The adsorption rate r_A and the desorption rate r_D of, e.g., N on a GaAs surface with Sb as a surfactant can be expressed as a function of the N partial pressure p_{N} as follows:

$$r_A = k_A^{\mathrm{N}} p_{\mathrm{N}} (1 - \theta_{\mathrm{As}} - \theta_{\mathrm{Sb}} - {}_{\mathrm{N}})$$

and

$$r_D = k_D^{\mathrm{N}} \theta_{\mathrm{N}} \quad \text{with constants} \quad k_A^{\mathrm{N}}, k_D^{\mathrm{N}} \tag{1}$$

At steady state it is assumed that the two rates will be equal. In this case the N content in GaAs:N grown with Sb can be written as

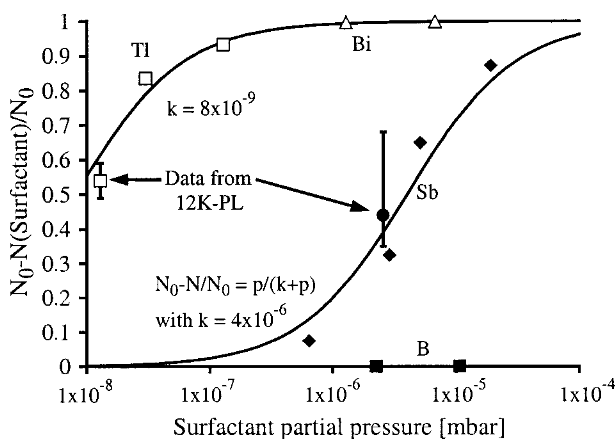

FIG. 4. Influence of the Tl Bi, Sb, and B partial pressure on the N content in GaAs:N layers grown under similar conditions. The data are plotted as the N concentration without surfactant (N_0) minus that with surfactant (N) normalized by the N content without surfactant. Data from SIMS analysis and 12 K Ph are shown. The curves correspond to the best fit of the data to the simple competitive adsorption model.

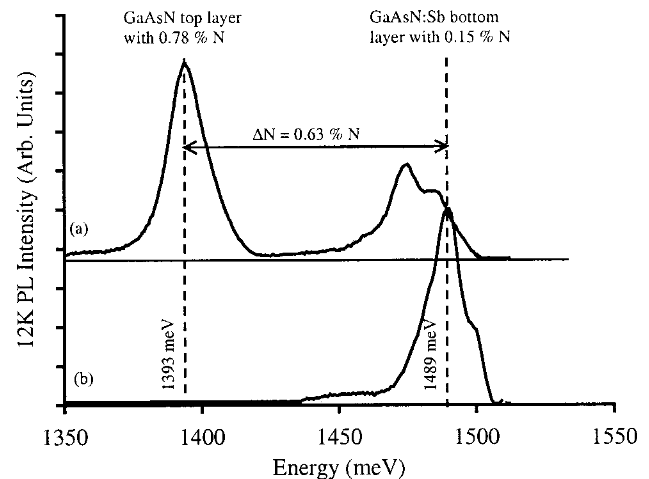

FIG. 5. 12 K photoluminescence of a GaAa:N double layer structure. The top layer was pure GaAs:N, whereas the bottom layer was grown with an Sb flow of TESb/TEGa = 6×10^{-2}. Spectrum (a) was measured on the as-grown sample and (b) was measured after removal of the top layer. This procedure allows identification of the PL peaks from the two layers.

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$$N_S = C\,\theta_N = C\cdot\frac{k_A^N p_N - k_A^N p_N \theta_{As}}{k_A^N p_N + k_D^N} - C\cdot\frac{k_A^N p_N}{k_A^N p_N + k_D^N}\cdot\theta_{Sb}\quad\text{with}\quad C=\text{constant}. \tag{2}$$

A calculation of the N content in GaAs:N without Sb shows that the first term in Eq. (2) is equal to the nitrogen concentration N_S^0 in GaAs:N grown without Sb and is, of course, independent of the Sb partial pressure. Therefore, one can write Eq. (2) as

$$\frac{N_S^0 - N_S}{N_S^0} = \frac{C\cdot\dfrac{k_A^N p_N}{k_A^N p_N + k_D^N}\cdot\theta_{Sb}}{C\cdot\left[\dfrac{k_A^N p_N - k_A^N p_N\theta_{As}}{k_A^N p_N + k_D^N}\right]} = \frac{\theta_{Sb}}{1-\theta_{As}} \tag{3}$$

The surface coverage θ_{Sb} with Sb can be written as a function of the Sb partial pressure and the surface coverage with As and N

$$\theta_{Sb} = \frac{k_A^{Sb}\cdot p_{Sb}(1-\theta_{As}-\theta_N)}{k_D^{Sb}+k_A^{Sb}\cdot p_{Sb}}. \tag{4}$$

Combining Eqs. (3) and (4), coupled with the assumption that the As surface coverage is much larger than the N surface coverage leads to the following expression for the dependence of N incorporation on Sb partial pressure:

$$\frac{N_S^0 - N_S}{N_S^0} = \frac{k_A^{Sb}\cdot p_{Sb}(1-\theta_{As}-\theta_N)}{(1-\theta_{As})(k_D^{Sb}+k_A^{Sb}p_{Sb})} \cong \frac{p_{Sb}}{k+p_{Sb}}\quad\text{with}\quad k = k_D^{Sb}/k_A^{Sb}. \tag{5}$$

The same model can be applied to any other group-V surfactant atom, such as Bi, competing with N on the GaAs surface. The equation was fitted to the measurements shown in Fig. 4, by varying the constant k. One can see that an excellent agreement of the theoretical model with the experimental behavior for Sb results.

For TESb the constant k in Eq. (5) was found to be 4×10^{-6}, whereas a much smaller value of k of 8×10^{-9} was calculated for Bi and also for Tl, a group-III atom. This is consistent with the reduced volatility of both Bi and Tl as compared to Sb. Thus, small Bi and Tl flows lead to a significant surface coverage and a concomitant reduction of the N incorporation in GaAs:N. In fact, a ratio of the partial pressures of $(C_5H_5)_2Tl$ and TEGa of only 0.0007 leads to an 85% reduction of the N content in GaAs:N.

The experimental data for Sb, Bi, and even Tl seem to be well explained by the Langmuir model, showing that the incorporation of N is strongly dependant on the surface coverage of these species. As Tl naturally sits on the group-III lattice, an explanation of the experimental results using the Langmuir model is unexpected. The group-III element In has also been reported to significantly reduce N incorporation in OMVPE grown GaAs:N.[11,16] Gas phase reactions between the Tl or In precursor and DMHy might be a possible explanation for the results. However, for $Ga_{0.5}In_{0.5}As$ Friedman *et al.* have shown that the In content is independent of the DMHy flow[16] making this argument unlikely. Other surface kinetic effects might be important. For example, a decrease in the sticking coefficients of N–C and N–H complexes by Tl and In species on the surface would explain the phenomena observed. It is also possible that Tl can adsorb on group-V sites. In addition, the bulky Tl and In atoms might influence the adsorption energy at a neighboring surface site and therefore affect the attachment of N complexes.

For In it is well known that the reduction of the N content in GaInAs:N depends on the N precursor and is not observed during CBE or MBE growth.[9,11] The same seems to be true for Sb. An increase in the N content with increasing Sb partial pressure was found for MBE grown material,[9] whereas this article reports a significant reduction of N. The different behavior might be due to the different source materials used and the low pressure applied during CBE growth. The volatile N complexes in OMVPE grown GaAs:N samples seem to be very sensitive to the group-III as well as group-V surface coverage during growth.

SUMMARY

This article reports on mechanisms for N incorporation in OMVPE grown GaAs:N layers with N content as large as 3%. A typical wedding cake morphology with steps oriented in the $[110]$ and $[\bar{1}10]$ directions was found for GaAs as well as GaAs:N at a growth temperature of 560 °C. The N incorporation on singular substrates is found to be the same for substrates with a small misorientation angle of 3° in either the $\langle 111\rangle A$ or B direction. A large misorientation angle of 6° toward $\langle 111\rangle A$ resulted in a significantly reduced N content. This was explained using a simple model of the bonding at $[110]$ and $[\bar{1}10]$ steps.

Sb, Bi, and Tl species on the surface during growth result in a reduction in the N incorporation into GaAs, whereas small B partial pressures were shown to have no effect. The behavior for Sb and Bi was well explained using a competitive adsorption model. The functional dependence of the N content on the surfactant partial pressure was perfectly explained by a Langmuir model.

Tl sits on the group-III lattice and, therefore, the competitive adsorption model would indicate that it would have no influence on the N incorporation in GaAs:N if Tl adsorbs only on group-III surface sites. Contrary to this prediction Tl addition does decrease the N content significantly, as has been reported for In as well. This behavior may be related to one of several factors including adsorption on group-V sites, interference with adsorption on group-V sites due to its large size, or factors associated with the DMHy N precursor such as a reduction of the sticking coefficient for volatile N–C and N–H complexes on the surface.

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