HTML ABSTRACT + LINKS

The use of nitrogen to disorder GaInP

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(Received 6 October 2003; accepted 3 March 2004)

Significant changes in microstructure, surface structure, and alloy composition have been observed in GaInP with the addition of nitrogen. These effects occur due to surface changes induced by small concentrations of nitrogen. Transmission electron microscopy and photoluminescence experiments indicate that the use of the surfactant N nearly eliminates the $CuPt_B$ ordered structure typically seen in organometallic vapor phase epitaxially grown $Ga_{0.5}In_{0.5}P$ lattice matched to GaAs. *In situ* surface photoabsorption measurements show a large change in the surface structure indicating that N reduces the concentration of P dimers on the surface, decreasing the driving force for ordering. Nitrogen also has a significant effect on the Ga/In ratio in the bulk. The indium content in the solid is decreased by 71% with a DMHy/III ratio of 60 (DMHy—1,1 dimethyl hydrazine). Nitrogen roughens the surface causing the formation of hillocks and small rectangular features that can be up to 50 nm tall. © *2004 American Institute of Physics.* [DOI: 10.1063/1.1715139]

I. INTRODUCTION

GaInP grown by organometallic vapor phase epitaxy (OMVPE) is used in many high efficiency optoelectronic devices, including light emitting diodes[1] and solar cells.[2] Considerable interest also exists for the use of GaInP/GaAs in heterojunction bipolar transistors.[3]

One of the interesting features of GaInP is that it exhibits CuPt ordering when grown using typical OMVPE conditions on (001) oriented GaAs substrates.[4] Alternating stresses resulting from $[\bar{1}10]$ oriented phosphorous dimers on the (2×4) reconstructed[5] (001) surface thermodynamically drive the atomic scale ordering observed in GaInP. The phosphorous dimers result in alternating {111} planes of Ga and In atoms. Typically, only two of the four possible variants of CuPt ordering are observed with ordering on the $(\bar{1}11)$ and the $(1\bar{1}1)$ planes,[6] the so-called $CuPt_B$ variants. CuPt ordering is dependent on growth conditions.[7] It is particularly significant for devices because it can decrease the band gap up to 160 meV for partially ordered material.[8] This large change in band gap can be controlled by varying the growth conditions.

Recently, much work has focused on using surfactants to control the surface during growth. Surfactants are surface active species that have a low solid solubility. Thus, they collect at the surface during growth, either covering the surface or at critical growth sites such as step edges. The surfactant induces changes in the growth process that can be either kinetic or thermodynamic. For example, Te has been shown to disorder GaInP by increasing the step velocity.[9] However, Sb acts differently to disorder GaInP by changing the surface reconstruction.[10] The effects of surfactants larger than and isoelectronic with P have been well documented.[11]

Surfactant effects are not limited to GaInP. Surfactants have been used in both elemental[12] and other compound semiconductor material systems.[13] Sb has been used in the growth of GaInAsN 1.3 μm lasers to improve material properties[14] as well as in the growth of AlGaAs to improve the optical quality.[15] The surface roughness of GaN[16] and of GaAsN and GaInAsN has been reduced with the use of Bi.[17]

Dilute nitride alloys are technologically important for use in high efficiency solar cells[18] and in 1.3–1.55 μm lasers.[19] The equilibrium solid solubility of N in III–V systems is expected to be extremely low.[20] However, due to surface effects, nonequilibrium phenomena yield much higher N concentrations using growth techniques such as molecular beam epitaxy (MBE) and OMVPE.[21] By adding small amounts of N into GaAs, the usefulness of GaAs materials may be extended. Because of the large bowing parameter associated with N, small amounts yield large changes in the band gap.[22] This allows materials to be grown lattice matched to GaAs with the addition of either In or Sb, but having band gaps in the near infrared.[23] The dilute nitride alloys have presented many challenges. Nitrogen incorporation has been found to depend significantly upon the nitrogen precursor in OMVPE growth[24] and upon the chemical composition of the alloy for both OMVPE (Ref. 25) and MBE (Ref. 26) growth.

Most of the research attention in dilute nitride alloys has focused on GaAs related materials. However, some work has been done on GaInPN because of its potential use in optoelectronic and electronic devices.[27,28] Holonyak *et al.* demonstrated the use of GaInP:N lasers using the nitrogen A line,[29] and several groups have investigated the band structure of nitrogen doped GaInP.[27,30] The effects of N addition during OMVPE growth on the surface properties and ordering of GaInP have not yet been explored.

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This paper presents the results of the use of N as a surfactant during the OMVPE growth of GaInP. Nitrogen is different from previous surfactants studied by this group (e.g., As, Sb, and Bi) because it is smaller than P and is much more volatile than previous surfactants. Because of its high volatility, higher partial pressures of the N precursor are required during growth to significantly alter surface properties. This does not lead to significant nitrogen incorporation but strongly influences surface properties and ordering in GaInP.

II. EXPERIMENT

For this study, GaInP:N epilayers were grown in an infrared heated, horizontal flow, atmospheric pressure OMVPE reactor equipped with optical windows for *in situ* surface photoabsorption measurements. Samples were grown on singular [nominally (001)], vicinal (3 °B misoriented), and (511) B GaAs substrates. Trimethylgallium (TMGa) and ethyldimethylindium (EDMIn) were the group III precursors with tertiary-butylphosphine (TBP) used for the phosphorous source. The nitrogen source was 1,1 dimethylhydrazine (DMHy). The carrier gas was Pd-diffused hydrogen and the total flow rate was 5200 SCCM. Growth was done at a temperature of 620 °C and growth rate of 1 μm/h. The TBP/III ratio was kept constant at 40 and the DMHy partial pressure was varied from 0 to 2.82×10^{-3} bars. Bilayer structures were grown with a GaInP layer followed by a layer grown with the surfactant nitrogen. A 3 min interrupt was used between the layers with TBP and DMHy flowing in the reactor but with no group III precursors. Typical layer thickness varied from 200–300 nm for the GaInP layer to up to 500 nm for the GaInP:N layer.

Surface photoabsorption (SPA) was used for *in situ* measurements of the surface structure. *P*-polarized light from a 150 W xenon lamp was used to irradiate the GaInP surface at an incidence angle of ≈70°, and the reflected light was dispersed through a monochromator and detected with a Si PNN$^+$ photodiode using standard lock-in techniques. SPA measurements were done at 620 °C and consisted of both wavelength and time scans. The wavelength scans were from 324 to 800 nm. Wavelength scans were done by measuring the reflectivity of a group V terminated surface (P dimers parallel to the light). The TBP was then removed from the system and a group III terminated surface was allowed to stabilize for 3 min and measured over the same wavelength range. To recover a group V terminated surface, TBP was added to the system and allowed to stabilize for 3 min. The process was then repeated with light incident in the $[\bar{1}10]$ direction (P dimers perpendicular to the light). Time scans were used to monitor the P dimer peak intensity at 410 nm.

Samples were characterized by x-ray diffraction (XRD) and photoluminescence (PL). PL was done at 20 K, excited with the 488 nm line of an Ar$^+$ laser with a power of 10 mW focused to a 0.5 mm^2 spot size. A SPEX 500 M spectrometer with the entrance and exit slits set to 200 μm was used in conjunction with a Hamamatsu head-on photomultiplier and standard lock-in techniques. Using the peak energy obtained from the PL measurements and the XRD peak from a $\theta/2\theta$ scan, the chemical composition was estimated for the GaInP:N layers using Vegard's law extended to quaternary systems. Secondary ion mass spectrometry (SIMS) was performed at Applied Microanalysis Laboratory using a Cameca ims-3f systems with a Cs$^+$ ion beam on one sample to verify the composition estimation procedure. The chemical composition was found to be within 3% of the estimation from XRD and PL. The degree of order (*S*) was calculated from the PL peak energy for the GaInP samples as[31]

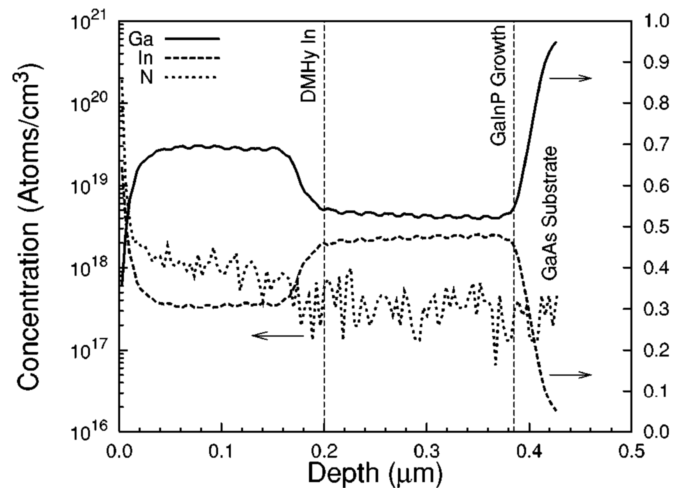

FIG. 1. SIMS depth profile of a GaInP:N/GaInP sample grown with DMHy/III=40. SIMS shows that nitrogen is only incorporated at dopant levels.

$$S=\sqrt{\frac{2005-\text{PL peak energy at 20 K}}{471}}\ \text{meV}. \quad (1)$$

Transmission electron microscopy (TEM) was used to verify the effect of nitrogen on ordering. For TEM experiments, two orthogonal ⟨110⟩ cross-section specimens were prepared using standard procedures and finished by Ar$^+$ ion thinning with the specimen cooled to ≈77 K. Bright field, dark field, and electron diffraction experiments were performed using a JEM 2010 instrument operated at 200 kV. The thicknesses of the thin foils examined by TEM were in the range of 150–400 nm with the examined regions thinned to a much smaller thickness. Energy dispersive x-ray spectroscopy (EDS) was used to characterize the interfacial regions to determine the chemical composition.

Surface morphology was studied using atomic force microscopy (AFM) with a Digital Instrument Dimension 3000 system. Scans were done in tapping mode using etched Si tips with a 5 nm radius. Scan size varied from under 1×1 μm^2 to 40×40 μm^2.

III. RESULTS

Nitrogen is not significantly incorporated into the GaInP layer. Figure 1 shows that nitrogen incorporation is small even for high (DMHy/III=40) partial pressures of DMHy. The SIMS depth profile shows that the N content is just above the detectability limit for the layer grown with DMHy. The N is incorporated only at dopant levels ($\approx 10^{18}$ cm^{-3}). However, with the introduction of DMHy, the Ga/In ratio in the solid is increased significantly. Figure 2 shows the decrease in In content in the solid with increasing DMHy/III. It

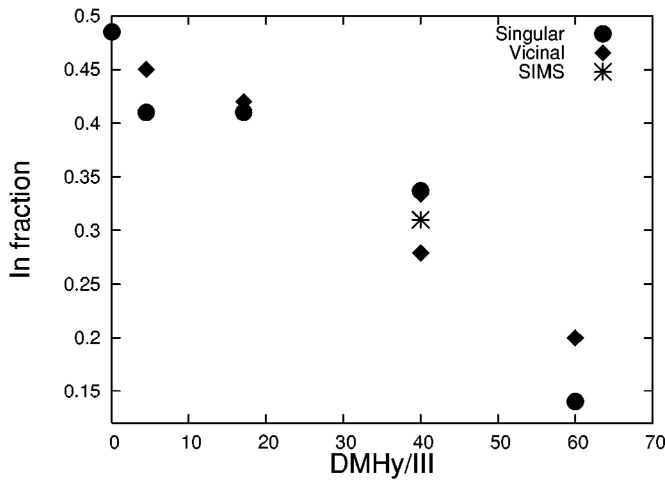

FIG. 2. In content in the solid vs DMHy/III ratio in the vapor for both singular and vicinal substrates.

appears not to be influenced by substrate orientation, but is dependent on the DMHy partial pressure. With the highest flow of DMHy, the In solid content is reduced by 71%. Samples were grown lattice matched to GaAs by increasing the EDMIn partial pressure.

Nitrogen also affects GaInP growth in ways that can be observed in the PL spectra of GaInP:N samples. Figure 3 is a PL spectrum for a GaInP:N/GaInP sample grown with DMHy/III=40 showing four peaks. The two most intense peaks correspond to the GaInP layer and the GaInP:N layer. The 1700 meV and 2270 meV peaks result from the addition of DMHy during GaInP growth. In order to determine the origin of these peaks, samples were etched in a 1:20:1 solution of $HCl:CH_3COOH:H_2O$. The 1700 meV peak disappeared only after the two highest energy peaks were no longer seen in PL. This indicates that it originates from a layer close to the undoped GaInP layer. High resolution TEM images confirm the presence of a 3–6 nm thick interfacial layer between the GaInP and GaInP:N layers. The high energy peak corresponds to a Ga-rich layer and shifts depending on the Ga/In ratio, disappearing when samples are lattice matched to GaAs.

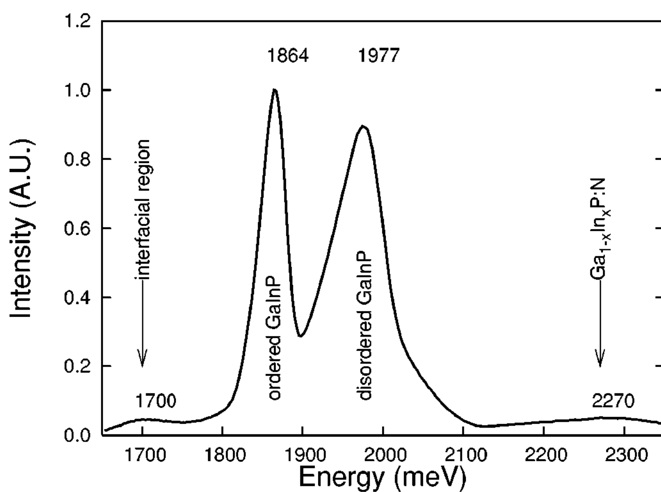

FIG. 3. PL of a GaInP:N/GaInP sample grown with DMHy/III=40.

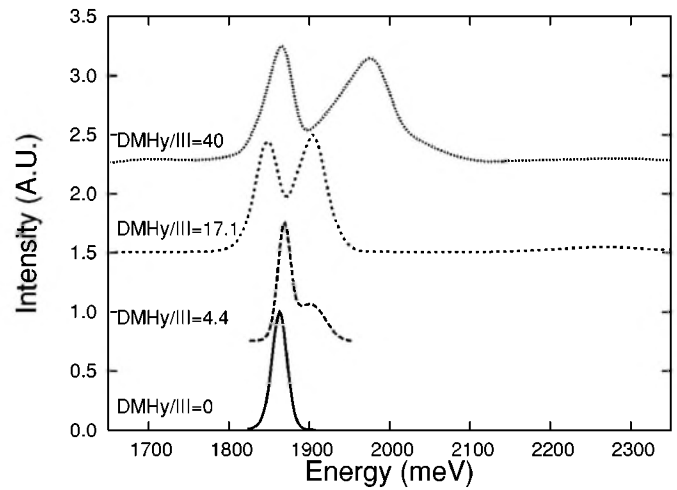

FIG. 4. 20 K PL spectra for bilayer structures of GaInP:N/GaInP grown with several DMHy/III ratios. The peak energy from the GaInP layer grown without DMHy remains virtually constant, while the peak energy from the GaInP:N layer is shifted to higher energies with increasing DMHy/III ratio.

The most important effect nitrogen has on GaInP is clearly observed from the PL data: Small amounts of N reduce the degree of CuPt ordering. At 20 K, the PL peak energy for undoped GaInP grown on nominally (001) GaAs is 1864 meV, corresponding to an order parameter (S) of 0.55, typical of GaInP grown under these conditions.[32] Figure 4 shows the effect of increasing DMHy/III ratio on the PL of GaInP. For a DMHy/III ratio of 4.4, a high energy shoulder emerges. As the DMHy/III ratio is increased, the strong lower energy peak corresponding to the ordered GaInP layer stays at approximately a constant energy, but a second peak, due to GaInP:N, emerges and shifts to higher energy for higher DMHy partial pressures. For DMHy/III =40, the GaInP:N is significantly disordered ($S=0.24$) and the order is almost completely eliminated ($S=0.14$) with a DMHy/III ratio of 60. Using samples grown lattice matched to GaAs, the dependence of order parameter on DMHy/III ratio from these data is plotted in Fig. 5.

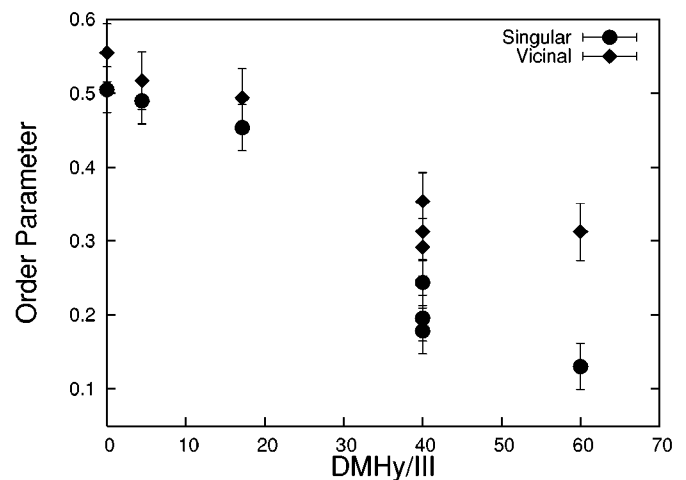

FIG. 5. Order parameter, deduced from the PL peak energy, as a function of DMHy/III ratio in the vapor.

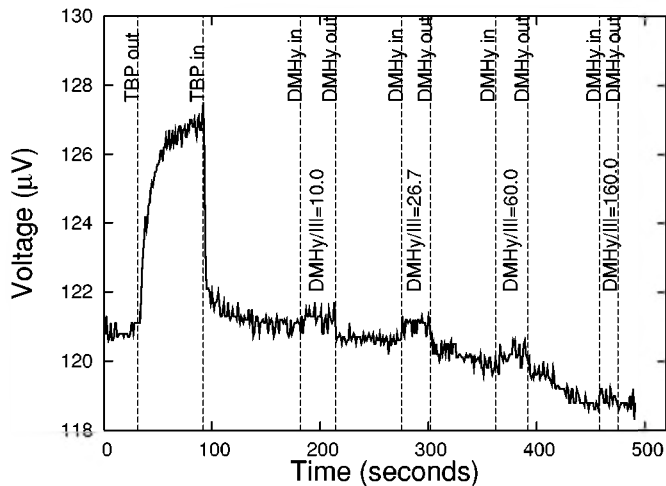

FIG. 6. SPA time scan of the P dimer peak at 410 nm with several DMHy/III fluxes. Increase in intensity corresponds to a decrease in P dimer concentration.

These PL measurements show that N disorders GaInP. SPA measurements were performed to understand how this occurs. Figure 6 shows the SPA intensity at 410 nm versus time from the P dimers oriented in the $[\bar{1}10]$ direction.[33] The intensity is inversely proportional to the $[\bar{1}10]$ P dimer concentration. When TBP is removed from the system ($30\,\mathrm{s}<t<90\,\mathrm{s}$), a group III terminated surface stabilizes and the signal intensity at 410 nm is a maximum. As TBP is once again introduced to the system ($t=90\,\mathrm{s}$), P dimers form on the surface and the signal at 410 nm decreases to the initial magnitude. As DMHy is added to the system, the signal again increases in magnitude. This corresponds to a decrease in surface coverage of $[\bar{1}10]$ P dimers.

The order parameter reduction deduced from PL is confirmed by the TEM results. A dark field TEM cross section of the GaInP/GaInP:N heterostructure grown on a 3 °B GaAs substrate shows a marked decrease in the order parameter, as seen in Fig. 7. Contrast in the image is dependent on the atomic scale ordering so that highly ordered regions are brighter. A 3 min interrupt of growth with only TBP and

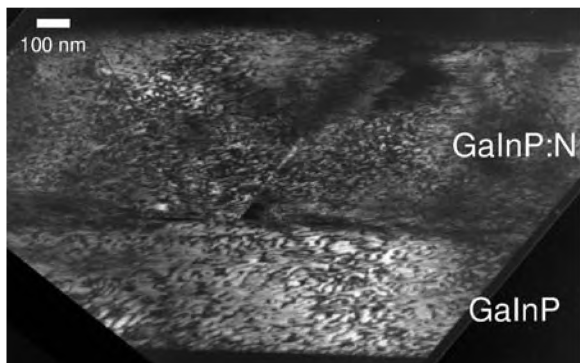

FIG. 7. Cross-sectional TEM dark field image using the $1/2(\bar{3}31)$ spot. Bright regions correspond to highly ordered material. The sample was grown lattice matched to GaAs on a vicinal substrate with a DMHy/III ratio of 40.

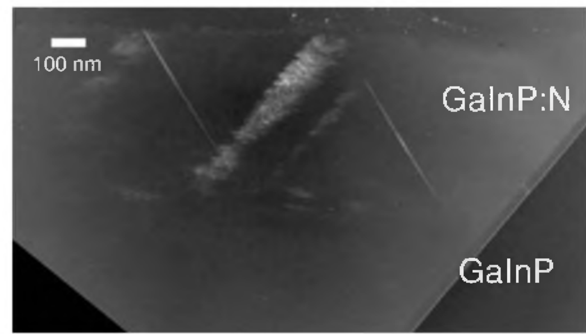

FIG. 8. Cross-sectional TEM dark field image using the $1/2(3\bar{3}1)$ spot for the same area as shown in Fig. 7. The second variant of CuPt_B ordering, normally not observed in 3 °B oriented samples, is observed.

DMHy flowing was used between the two layers. It is seen that the GaInP:N layer is quickly disordered once growth is resumed.

Another interesting feature shown in the TEM images is the emergence of a second variant of CuPt_B ordering in the vicinal sample. Since all the steps on 3 °B misoriented substrates move in the same direction, the $(1\bar{1}1)$ variant of CuPt ordering is normally the only observed variant for GaInP grown on B misoriented substrates. However, with the addition of N, regions of the other variant, CuPt_B, $(\bar{1}11)$, can be seen in Fig. 8.

AFM scans indicate that the surface is roughened by the addition of N and that large hillocks appear on the surface. Figure 9 is an AFM micrograph of a $10\times10\ \mu\mathrm{m}^2$ area of a GaInP:N sample grown with DMHy/III=40 on a 3 °B misoriented (vicinal) substrate. In addition to the hillocks, small rectangular features appear. These features can be over 40 nm in height and have steep sidewalls with angles greater than the sidewall ($>35°$) of the AFM tip.

IV. DISCUSSION

The SIMS depth profile for the GaInP:N sample indicates that N incorporation in GaInP lattice matched to GaAs is extremely small. Previous studies have indicated that substrate orientation influences N incorporation: A-oriented steps increase N incorporation and B-oriented steps have no effect.[34] Our results for N incorporation on nominally (001), 3 °B misoriented, and (511) B GaAs substrates show no difference in N content for epilayers grown on the different substrates, agreeing with previous studies indicating that B-oriented steps do not influence N incorporation.

The solid solubility of N in GaInP is expected to be low as it is for most III–V systems.[20] However, nitrogen incorporation in GaAs is much higher than thermodynamically predicted.[22,25,35] For indium compounds much smaller N concentrations are observed.[36,37] It is unknown exactly why In inhibits N incorporation but the relatively weak In-N bond may be partly responsible.[25] Regardless of the mechanism for low N incorporation, the use of nitrogen as a surfactant is one more tool for controlling the surface during growth of GaInP. Since nitrogen is not incorporated significantly and influences the surface, as seen in SPA, nitrogen behaves as a surfactant in the growth of GaInP. Nitrogen is different from

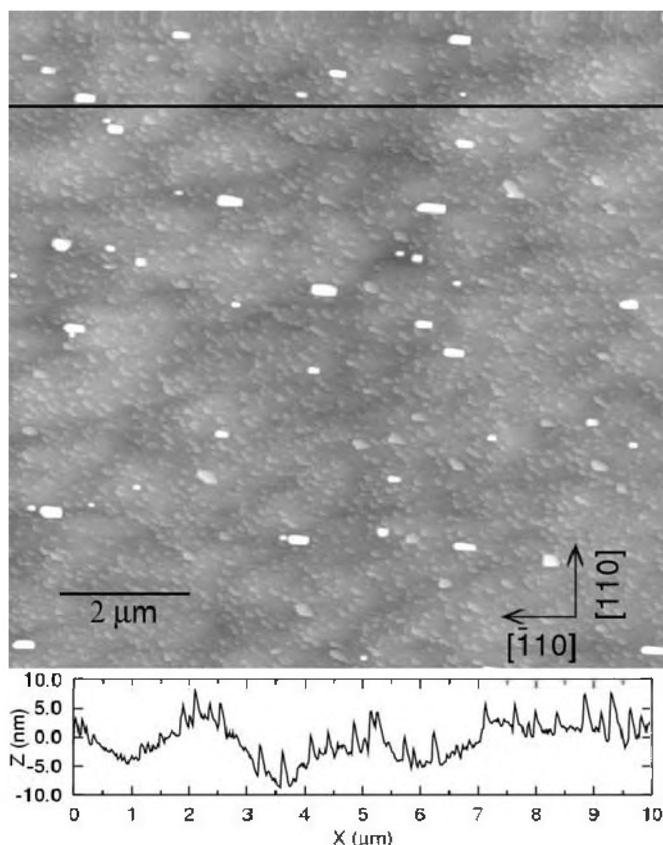

FIG. 9. AFM image of GaInP:N grown with DMHy/III=40. Surfaces become rougher with the addition of DMHy. The Z scale is from 0 (black) to 50 (white). The black line near the top of the figure is where the cross section, shown below, was taken.

other surfactants in that it is highly volatile. This high volatility requires large partial pressures to produce significant effects; however, the addition of N during GaInP growth changes the surface and reduces the degree of order. Thus, N clearly acts as a surfactant.

Figure 1 also shows an increase in the Ga/In ratio. TMGa and EDMIn flows were kept constant from the undoped GaInP layer to the GaInP:N layer; however, the sample becomes Ga rich. The change in Ga/In ratio is thought to be a result of parasitic gas phase reactions between the EDMIn and DMHy. Some of the reduction may also be due to In evaporation from the surface as is seen in InGaN.[38] Other groups have not observed a noticeable change in Ga/In ratio when growing III-V-N materials.[25,35] The differences in results may be due to the reduction in parasitic reactions under low pressure growth conditions. The In fraction is steadily reduced with increasing DMHy flow and is decreased by 71% for the highest DMHy partial pressure (DMHy/III=60). It is thought that gas phase parasitic reactions provide the dominant mechanism for a reduction in the In solid composition because of the magnitude of the decrease and because there is no discernible effect from substrate misorientation. If the reduction in In content is from parasitic reactions one would not expect substrate orientation to influence the In content, but if the reduction in In were from In desorption from the surface, misorientation would be expected to have a noticeable effect.[39]

Nitrogen clearly reduces the degree of CuPt ordering. The mechanism by which nitrogen destroys ordering is postulated to be a decrease in the [$\bar{1}$10] P dimer surface concentration as deduced from SPA measurements. As DMHy is added to the system, the reflected intensity at 410 nm increases, due to a decrease of the [$\bar{1}$10] P dimer concentration on the surface. Nitrogen competes for surface sites with P. If nitrogen dimerized, one would expect the degree of CuPt ordering to increase because N dimers are smaller than P dimers, resulting in an increase in driving force for ordering. Ordering clearly does not increase, as shown by the TEM and PL data. This indicates that N does not dimerize on the GaInP surface. This observation is consistent with results for GaN: Nitrogen dimers have not typically been observed in GaN surface reconstructions. Both theoretical calculations[40] and experimental results[41] for GaN indicate that N-rich surfaces are (1×1) reconstructed. For MBE growth of cubic GaN on (001) GaAs under N-rich conditions the surface is also observed to have the (1×1) reconstruction.[42,43] This is because the strain energy to form N dimers is larger than the energy to form an unreconstructed surface. In GaInP, with a significantly larger atomic spacing, the strain energy of N dimers would be even larger.

It has been postulated that disordering of GaInP by nitrogen is caused by the reduction of P dimers on the surface, but a second possibility exists. Disordering in GaInP from the addition of N might be due to surface roughening. Nitrogen was shown to roughen the surface in GaAs for both OMVPE[44] and organometallic MBE[45] growth conditions. The change in surface morphology has been attributed to a decrease in the Ga adatom diffusion length. This work shows that the addition of N also roughens the GaInP surface: Hillocks are formed on the surface. The surface roughness increases with increasing DMHy/III ratio and may lead to the formation of many high angle facets on the sides of the hillocks. Misorientation angles exceeding 6° have been reported to decrease ordering.[32] This is the mechanism by which Br is believed to disorder GaInP.[46]

Substrates misoriented toward the B direction exhibit only one variant of CuPt ordering. As mentioned above, misorientation results in all the steps on the surface moving in the same direction. Since all the steps move in the same direction, only one variant is formed.[6] Figures 7 and 8 show the same physical area of the sample but from opposite poles. These dark field images are sensitive to ordering. Bright regions indicate areas of high ordering. When imaging with the 1/2($3\bar{3}1$) spot in Fig. 8, the undoped GaInP layer is dark as expected; however, bright regions appear in the GaInP:N layer indicating regions of the second variant of $CuPt_B$ ordering. AFM data in Fig. 9 show that the surface is roughened with the addition of N. Hillocks are formed that lead to the creation of steps moving in the opposite direction. With steps moving in the opposite direction, the second variant of $CuPt_B$ ordering is formed, even for growth on vicinal substrates.

Nitrogen produces complex photoluminescence spectra in GaInP as seen in Fig. 3. The PL spectra from a GaInP:N/GaInP sample show four peaks. The two strongest peaks correspond to the two layers grown, the highly ordered

GaInP layer and the less ordered GaInP:N layer. In addition, both a low energy and a high energy peak are seen. The high energy peak corresponds to a Ga-rich region that is ≈70% Ga. This peak is observed to shift in energy depending on the Ga content in the layer. For lattice matched samples, the high energy peak is not seen. The low energy peak is harder to explain. It was mentioned above that the low energy peak is attributed to a thin interfacial region that is formed between the GaInP and GaInP:N layers. Three possible explanations could account for the low energy peak.

(1) The interfacial region could be highly ordered. This is unlikely because the order parameter would have to be greater than 0.8 to explain the peak energy, and this trend is not seen in epilayers grown with nitrogen.

(2) The layer could contain several percent N. This is supported by results in GaAsN where an interfacial region of approximately the same thickness has been observed during OMVPE growth.[47] It was found that the interfacial layer had twice the N content of the epilayer.

(3) The interfacial layer is In rich. EDS data taken across the interfacial region indicate that the layer is, indeed, In rich. Furthermore, no increase in the N content is observed across the interface. Thus, we conclude that the low energy peak originates from an In rich interfacial region. The mechanism behind the formation of the In rich interface is not understood and is still under investigation.

V. CONCLUSIONS

The use of the surfactant N in the growth of GaInP is observed to cause a reduction in CuPt ordering. The reduction in order parameter is due to a change of the surface thermodynamics. Adding N to the surface produces an observable decrease in the $[\bar{1}10]$ P dimer concentration. The reduction of P dimer concentration on the surface reduces the driving force for ordering and, thus, reduces the amount of CuPt order in GaInP. Nitrogen, in sufficiently high quantities, is found to nearly destroy all of the CuPt order in GaInP.

Nitrogen also reduces the In content in the epilayer through gas phase parasitic reactions. Lattice matched layers were grown by increasing the EDMIn partial pressure. The addition of N during growth of GaInP also forms a thin In-rich interfacial layer with a PL peak at 1700 meV. Surface roughness is increased with the addition of N causing large hillocks to form. These hillocks allow the second $CuPt_B$ variant to form in samples grown on substrates misoriented in the B direction.

ACKNOWLEDGMENTS

The authors from the University of Utah wish to acknowledge the Department of Energy, Division of Basic Sciences, for their support in funding the research presented in this paper. T.Y.S. wishes to acknowledge the Korean Ministry of Education for financial support of his work.

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