Effects of V/III ratio on ordering in GaInP: Atomic scale mechanisms

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$Ga_{0.5}In_{0.5}P$ layers have been grown by organometallic vapor-phase epitaxy using various values of input V/III ratio for two phosphorus precursors, phosphine, the conventional precursor, and tertiarybutylphosphine (TBP), a newly developed, less-hazardous precursor. For growth on nominally (001) GaAs substrates misoriented by 3° (and in some cases by 0° or 6°) to produce [110] steps on the surface at a growth temperature of 620 °C, the Cu–Pt-type ordering is found to be strongly affected by the input flow rate of the phosphorus precursor (V/III ratio). For decreasing input partial pressures below 3 Torr for PH_3 and 0.75 Torr for TBP the low-temperature photoluminescence (PL) peak energy increases indicating a lower degree of order. This is confirmed by transmission electron diffraction results. The decrease in the degree of order corresponds to a decrease in the concentration of $[\bar{1}10]$-oriented P dimers on the surface, as indicated by surface photoabsorption spectroscopy results. These data indicate that the reduction in ordering is caused by the loss of the (2×4) reconstructed surface during growth. The difference in the behavior for PH_3 and TBP is interpreted as due to the lower pyrolysis efficiency of PH_3. The surface structure measured using high-resolution atomic force microscopy indicates that the [110] steps produced by the intentional misorientation of the substrate are bunched to produce supersteps approximately 30–40 Å in height for the lowest V/III ratios. The step height decreases markedly as the input phosphorus partial pressure increases from 0.4 to 0.75 Torr for TBP and from 1 to 3 Torr for PH_3. This corresponds to a change from mainly monolayer to predominantly bilayer steps in the vicinal regions between bunched supersteps. Stabilization of the bilayer steps is interpreted as due to formation of the (2×2) reconstruction on the $(111)B$ step edges. The degree of order is an inverted U-shaped function of the flow rate of the phosphorus precursor. Thus, use of very high input V/III ratios is also found to reduce the degree of order in the $Ga_{0.5}In_{0.5}P$ layers. These high input phosphorus flow rates are found to result in a monotonic increase in the density of $[\bar{1}10]$-oriented P dimers on the surface. This decrease in order is believed to be related to a change in the structure of kinks on the [110] steps at high V/III ratios. © *1996 American Institute of Physics.* [S0021-8979(96)07209-1]

I. INTRODUCTION

$Ga_{0.5}In_{0.5}P$ lattice matched to GaAs is an important III/V alloy used for both photonic devices, such as visible light-emitting diodes[1] and lasers,[2] and for electronic switching devices such as heterojunction bipolar transistors.[3] This material is most frequently grown by organometallic vapor-phase epitaxy (OMVPE) using trimethyl group-III precursors and PH_3. More recently, a movement has begun to replace the highly dangerous hydride by the much safer alternative, tertiarybutylphosphine (TBP).[4,5]

An important feature of this alloy is atomic-scale ordering during epitaxial growth to form the Cu–Pt structure, with ordering on the {111} planes.[6] Formation of this natural superlattice structure has a profound influence on the materials properties. For example, the band-gap energy of disordered $Ga_{0.5}In_{0.5}P$ is found to shrink by magnitudes as large 160 meV due to Cu–Pt ordering.[7] Thus, ordering must be controlled in materials to be used for device fabrication. The degree of order is found to vary widely for different growth conditions. For example, growth rate,[8] substrate temperature during growth,[9] and substrate misorientation[10] can be chosen in such as way as to eliminate ordering during OMVPE growth. An additional parameter, the input flow rate of the phosphorus precursor (or the V/III ratio), is also found to have a significant effect on the degree of order. The degree of order is found to be a maximum for a particular optimum V/III ratio and to decrease for both high and low V/III ratios.[11,12]

The mechanism for the effect of the V/III ratio on the extent of formation of the Cu–Pt structure during OMVPE growth is still undetermined. Kurtz *et al.*[12] postulated that the reduction in ordering for low V/III ratios was due to an alteration in the surface structure and at high V/III ratios the reduction in ordering was due to a postulated decrease in the group-III surface diffusion coefficients.

Based largely on theoretical evidence, it is widely accepted that the Cu–Pt structure is stable only near the surface when it is reconstructed to form the (2×4) structure consisting of $[\bar{1}10]$-oriented phosphorus dimers running in [110] rows on the surface.[6] For example, energy minimization calculations by Zhang, Froyen, and Zunger[13] indicate that the periodic surface stresses resulting from the formation of [110] phosphorus dimer rows on the (2×4) reconstructed surface result in a segregation of the subsurface group-III atoms into alternating [110] rows of In and Ga atoms. This,

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in turn, results in the formation of the two B variants of the Cu–Pt structure with ordering on the $(\bar{1}11)$ and $(1\bar{1}1)$ planes typically observed for GaInP layers grown by OMVPE.

Until recently, no information concerning the chemical structure of the surface during OMVPE growth was available. The optical technique of surface photoabsorption (SPA) has been demonstrated to reveal information about the bonding at the surface that can be interpreted to yield information about the surface reconstruction for both GaAs[14] and InP.[15] Of course, optical techniques are incapable of revealing the long-range order on the surface, so our knowledge of the actual surface reconstruction during OMVPE growth remains incomplete. Nevertheless, Murata *et al.*[16] demonstrated a direct correlation between the concentration of the $[\bar{1}10]$-oriented phosphorus dimers characteristic of the (2×4) reconstructed surface, detected using SPA, and the degree of order as the temperature was varied during the OMVPE growth of $Ga_{0.5}In_{0.5}P$.

Another characteristic of the surface related to ordering is the step structure. Several models of ordering in III/V alloys have invoked the motion of monatomic [110] steps on the surface to explain the strong influence of substrate orientation on ordering.[17–19] Again, until recently, no evidence of the actual surface structure has been available. Since the surface is rapidly oxidized after the cessation of OMVPE growth, techniques suitable for ultrahigh-vacuum systems such as scanning tunneling microscopy (STM) are not applicable. Fortunately, it has recently been discovered that the structure of the surface can be determined by examination of the oxidized surface using atomic force microscopy (AFM). The highly conformal nature of the oxide allows the detection of monolayer steps on the semiconductor surface.[20] This approach has been demonstrated to also reveal the atomic structure of the surface for GaInP layers grown by OMVPE.[21–23] For exactly (001)-oriented substrates, the surface of GaInP layers grown using the trimethyl group-III alkyls combined with phosphine at 670 °C with an input V/III ratio of 160 has been reported to consist of small islands surrounded by bilayer steps.[24]

The purpose of this article is to report the results of an experimental exploration of the mechanisms resulting in the dependence of order on input V/III ratio for GaInP layers grown by OMVPE on substrates misoriented to produce [110] steps on the surface. SPA and AFM techniques are used to probe the nature of the surface of layers grown using two phosphorus precursors, phosphine, and TBP, over a wide range of input partial pressures. The ordering is characterized using a combination of low-temperature photoluminescence (PL) and electron microscopy. The loss of order at low V/III ratios is found to correspond precisely to the marked reduction in the SPA signal attributed to the $[\bar{1}10]$ P dimers on the surface.

II. EXPERIMENT

The $Ga_{0.5}In_{0.5}P$ layers were grown by OMVPE on semi-insulating GaAs substrates typically misoriented by an angle of 3° toward the $[\bar{1}10]$ direction. A few runs were made on substrates with misorientation angles of 0° and 6°. The flow rates of the group-III source materials trimethylgallium (TMGa) and trimethylindium (TMIn) were held constant for all runs to give a solid lattice matched to the GaAs substrates and a growth rate of 0.5 μm/h.The V/III ratio was varied by changing the flow rate of the phosphorus precursor, either TBP, held in a temperature-controlled bath at −8 °C, or PH_3. Substrate preparation consisted of degreasing followed by a 1 min etch in a solution of $NH_4OH:H_2O:H_2O_2=2:12:1$. A horizontal, atmospheric pressure OMVPE reactor was used. A thin GaAs buffer layer was grown at 620 °C before initiating growth of the GaInP layer. The growth temperature was constant at 620 °C for all layers. The input partial pressure of the phosphorus precursors was varied from 0.75 to 12 Torr, corresponding to V/III ratios of 40–640, for PH_3 and from 0.094 to 3 Torr, corresponding to V/III ratios of 5–160, for TBP.

The solid composition of the GaInP layers was determined using Vegard's law, from x-ray-diffraction measurements using Cu $K\alpha$ radiation. The 20 K PL was excited with the 488 nm line of an Ar^+ laser. The emission was dispersed using a Spex Model 1870 monochromator and detected using a Hamamatsu R1104 head-on photomultiplier tube. [110] cross-sectional transmission electron microscope (TEM) samples were prepared by cleaving two facets, glued face to face and mechanically polished, followed by Ar-ion milling to electron transparency at 77 K. The transmission electron diffraction (TED) patterns and TEM images were obtained using a JEOL 2000 FXII scanning transmission electron microscope operated at 200 kV. The characterization of the surface structure was carried out using a Nanoscope III atomic force microscope (AFM) in the tapping mode. Etched single-crystalline Si tips were used with an end radius of about 5 nm, with a sidewall angle of about 35°. Scan rates of 1–2 lines per second were used and data were taken at 12 points/line and 512 lines per scan area. A SPA system attached to the OMVPE system was used for *in situ* measurements of the surface structure during growth. P-polarized light from a 150 W Xe lamp irradiated the GaInP layers at an incident angle of 70° through a polarizer and a chopper. The direction of the incidence light was parallel to the direction of gas flow in the reactor. The reflected light was monochromatized and detected by a Si PNN^+ photodiode using standard lock-in amplification techniques.

III. RESULTS

A. Growth using PH_3

Figures 1 and 2 show AFM images and section scans of GaInP surfaces for GaAs substrates misoriented by 3° in the $[\bar{1}10]$ direction for two PH_3 partial pressures (V/III ratios). Clearly, the [110] steps produced by the intentional misorientation of the substrate are bunched. From the section scans in Fig. 2, each superstep is seen to consist of an (001) facet on the left-hand side and a $(11n)$ facet on the right-hand side. The left-hand side facet is identified as (001) from the measured angle of $3°\pm0.1°$ between the facet and the nominal substrate surface. The surface consists of three "phases," (001) facets, the $(11n)$ facets, and the relatively large vicinal regions in between. The heights of the largest supersteps are plotted versus the V/III ratio in Fig. 3. Superstep heights

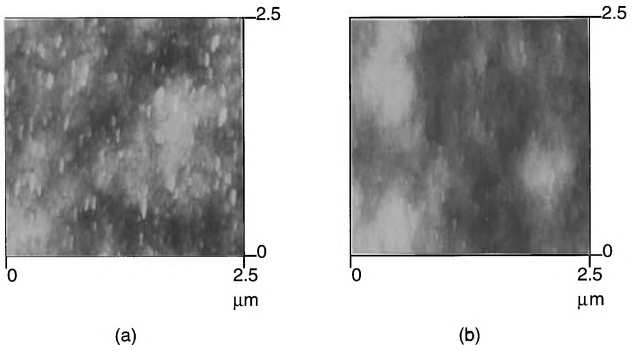

FIG. 1. AFM images of the surface structure for $Ga_{0.5}In_{0.5}P$ layers grown at 620 °C on GaAs substrates misoriented by 3° in the [$\bar{1}$10] direction with two PH_3 partial pressures. (a) V/III=80; (b) V/III=640.

as large as 30–40 Å are produced for the lower V/III ratios of 60 and 80. Even here the fraction of steps collected into supersteps is less than 0.2, i.e., 80% of the surface is vicinal. As the V/III ratio increases the stepbunching becomes less severe and the surface is comprised nearly entirely of the vicinal phase. For substrates with exactly the (001) orientation, step bunching is not observed;[24] however, for high V/III ratios, the individual steps are found to be clearly defined bilayers, approximately 5.7 Å in height. For low V/III ratios, the bilayer steps disappear and only monolayer (2.8 Å) steps are observed.[25,26] In Fig. 2, there is a hint that the same

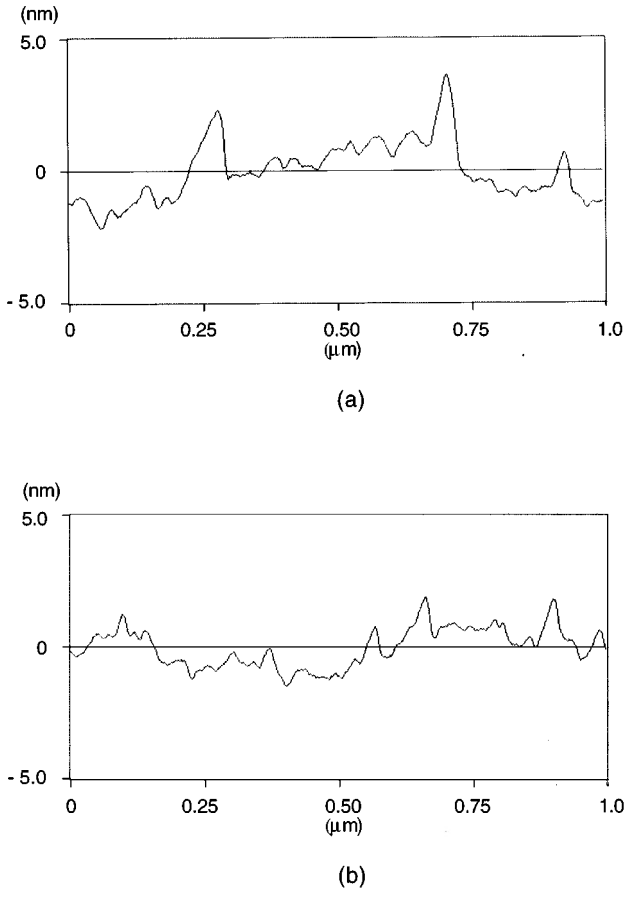

FIG. 2. AFM section scans of the surface structure for $Ga_{0.5}In_{0.5}P$ layers imaged in Fig. 1. (a) V/III=80; (b) V/III=640.

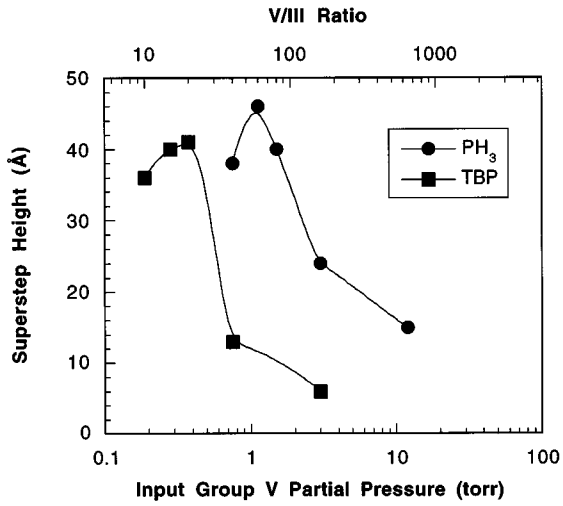

FIG. 3. Superstep height plotted vs input phosphorus partial pressure (V/III ratio) for PH_3 (●) and TBP (■).

phenomenon occurs for 3° misoriented substrates. Bilayer steps appear to occur with a greater frequency for the layer grown with a V/III ratio of 640. The disappearance of step bunching appears to coincide approximately with the formation of bilayer steps in the vicinal regions.

[110] pole TED patterns for the GaInP layers grown with several V/III ratios are shown in Fig. 4. The TED patterns show order-induced superspots due mostly the 1/2($\bar{1}$11) variant for all samples. This is consistent with previous reports[10,27,28] for a substrate misorientation of 3° where the ordered regions were found to be essentially all of the same

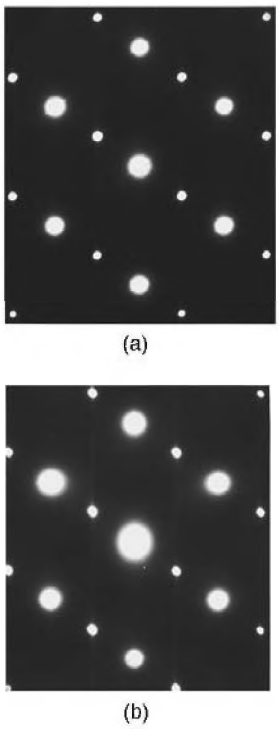

FIG. 4. [110] transmission-electron diffraction patterns obtained for $Ga_{0.5}In_{0.5}P$ layers grown at 620 °C using PH_3 with two V/III ratios. (a) V/III=40; (b) V/III=160.

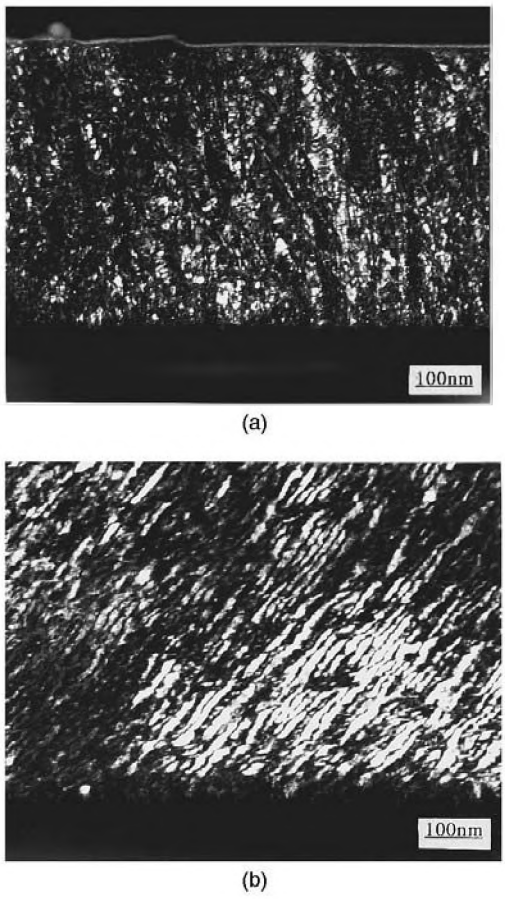

FIG. 5. Dark-field transmission electron microscope images of the $Ga_{0.5}In_{0.5}P$ layers with diffraction patterns shown in Fig. 4. (a) V/III=40; (b) V/III=160.

variant. Figure 5 shows typical (110) cross-sectional dark-field electron microscope images obtained for the $Ga_{0.5}In_{0.5}P$ layers used to obtain the diffraction patterns in Fig. 4. A stripe contrast pattern covers most of the area in the photographs. Dispersed, platelike superlattice domains are observed for GaInP grown with a V/III ratio of 40. The small domain size precludes using the intensities of the order-induced spots as an indication of the degree of order.[10]

Figure 6 shows the PL peak energies at 20 K for excitation by the 488 nm line of an Ar^+ laser with a power of 10 mW. The minimum peak energies for all substrate misorientations occur for a PH_3 partial pressure of 3 Torr (V/III =160). The degree of order decreases dramatically for lower V/III ratios, below the optimum, and more gradually with increasing V/III ratios, above the optimum.

B. Growth using TBP

Results very similar to those reported above for layers grown using PH_3 were obtained using TBP. The PL peak energies for the samples grown using TBP are plotted along with the PH_3 data in Fig. 6. The general features of the data for TBP are remarkably similar to those for the samples grown using PH_3, although the minimum PL peak energy is higher for the samples grown using TBP. The effects occur at lower input partial pressures for the TBP. This difference

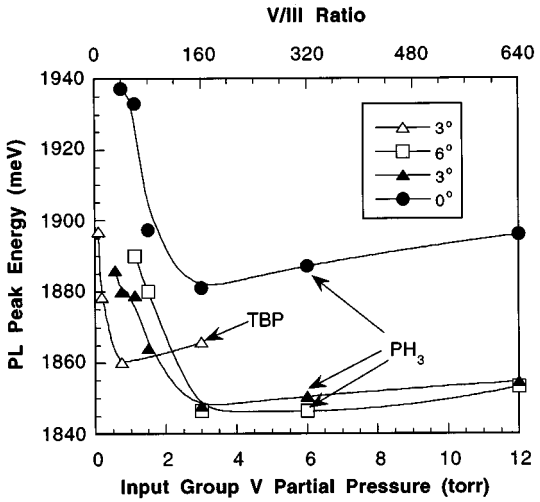

FIG. 6. 20 K PL peak energy vs input phosphorus partial pressure for various substrate misorientations.

is attributed to the difference in the extent of decomposition for the two precursors. The data for the two precursors exhibit identical trends when the PH_3 partial pressure is divided by a factor of 4.

Figures 7 and 8 show AFM images and sectional scans, respectively, for GaInP layers grown on GaAs substrates misoriented by 3° in the $[\bar{1}10]$ direction using two TBP partial pressures (V/III ratios). As for PH_3, the [110] steps are bunched to produce supersteps for low V/III ratios and the supersteps are suppressed at high V/III ratios. The superstep height is plotted versus the TBP partial pressure in Fig. 3. The trends for the two phosphorus precursors are nearly identical, although approximately 4× higher concentrations are required to suppress the supersteps for PH_3. As discussed below, this is thought to be due to the incomplete pyrolysis of PH_3 at 620 °C. As for PH_3, the vicinal region of the surface for the sample with the high V/III ratio [Fig. 8(b)] appears to consist mainly of bilayer steps.

Figure 9 shows the effect of input TBP partial pressure on the 400 nm SPA signal difference between the [110] and $[\bar{1}10]$ directions on the surface. This spectral feature is thought to be due to $[\bar{1}10]$-oriented P dimers.[15,16,25] It is clear that the SPA signal difference increases with increasing TBP

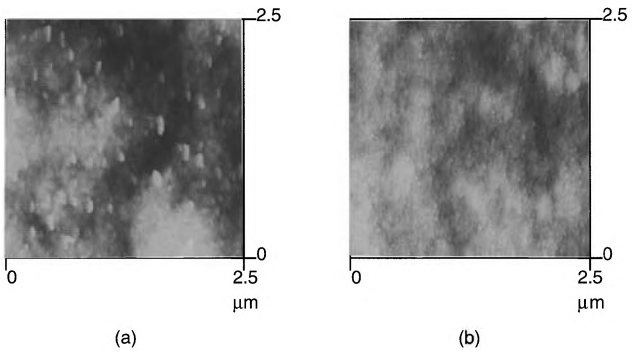

FIG. 7. AFM images of the surface structure for $Ga_{0.5}In_{0.5}P$ layers grown at 620 °C on GaAs substrates misoriented by 3° in the $[\bar{1}10]$ direction with two TBP partial pressures. (a) V/III=20; (b) V/III=160.

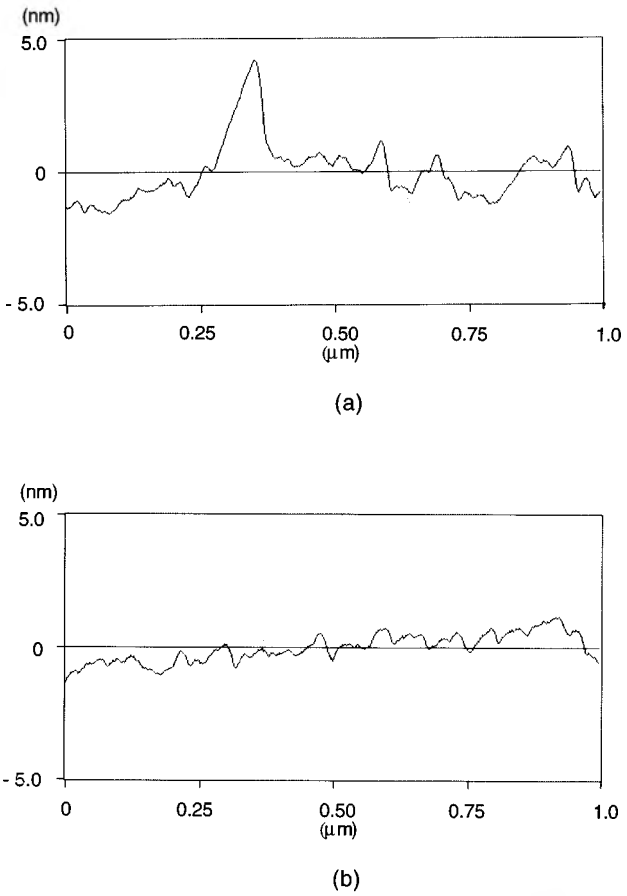

FIG. 8. AFM section scans of the surface structure for $Ga_{0.5}In_{0.5}P$ layers imaged in Fig. 7. (a) V/III=20; (b) V/III=160.

partial pressure at 620 °C, indicating that the concentration of the [$\bar{1}$10] P dimers characteristic of the (2×4) reconstructed surface increases with increasing TBP partial pressure.

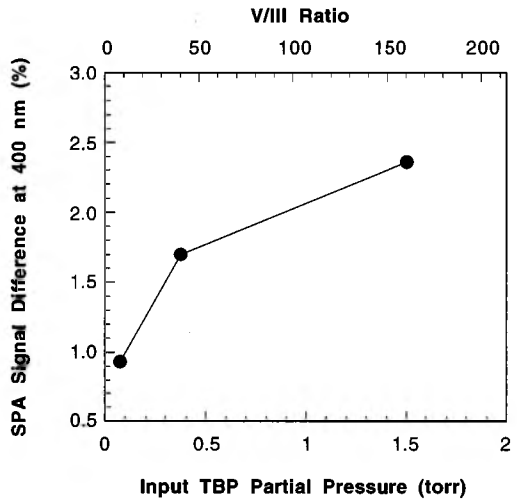

FIG. 9. TBP partial pressure dependence of SPA signal difference between [$\bar{1}$10] and [110] directions at 400 nm for GaInP layers grown on GaAs substrates misoriented by 3° in the [$\bar{1}$10] direction with various V/III ratios.

IV. DISCUSSION

A cursory overview of the results presented in the previous section shows that the phosphorus partial pressure (pyrolyzed TBP and PH_3 concentrations, denoted p_P^*) during OMVPE growth has a dramatic effect on both the surface structure (step bunching and surface reconstruction) and the materials properties, particularly the degree of order.

A comparison of the results obtained using PH_3 and TBP shows the same general effects on both step bunching and the degree of order, inferred from the PL peak energy. As discussed above, dividing the PH_3 partial pressure by a factor of 4 makes the effects of the partial pressure of the phosphorus precursor virtually the same for PH_3 and TBP. This is attributed to the much slower pyrolysis rate for PH_3, as indicated by published pyrolysis data.[29] It is difficult to make a quantitative comparison for the pyrolysis data, to see if the factor of 4 is appropriate, due to the strong effects of both the substrate and the presence of the group-III precursors.[29] The pyrolysis of TBP is probably virtually complete for the growth conditions and reactor geometry used in these experiments. The assertion that the PH_3 is only 25% pyrolyzed is in qualitative agreement with the published pyrolysis data.

The experimental data indicate that the effects of V/III ratio on the SPA signal, the step bunching, and the PL peak energy occur virtually in tandem for p_P^* values between 0.1 and 1 Torr for both precursors. The increase in the SPA signal corresponds to an increase in the concentration of [$\bar{1}$10]-oriented P dimers on the surface caused by an increase in the percentage of the surface terminated by P as p_P^* is increased. The presence of these dimers is indicative of a (2×4) reconstruction at the surface. Thus, we conclude that the percentage of the surface covered by the (2×4) reconstruction increases with increasing p_P^*. The corresponding increase in the degree of order, deduced from the decrease in the PL peak energy and the TED results, indicates that formation of the (2×4) reconstructed surface leads to production of the Cu–Pt ordered structure, as suggested by theoretical energy minimization calculations.[13] This is consistent with our previous results showing that the decrease in the concentration of the [$\bar{1}$10]-oriented P dimers with increasing growth temperature for a constant V/III ratio corresponds to a reduction of Cu–Pt order with increasing growth temperature for GaInP growth on exactly (001) GaAs substrates using TBP.[16]

This interpretation of the experimental data would indicate that the increase in [110] step bunching for misoriented substrates at low values of p_P^* is an incidental byproduct of the change in the chemical structure of the surface with changing V/III ratio. The disappearance of the supersteps may reflect the change to a (2×4) reconstructed surface on the (001) facets at high V/III ratios, although the mechanism for such an effect is unclear. Much more likely is that the decrease in step bunching is related to the change in stability of the bilayer steps due to reconstruction of the step riser. For a monolayer step, no reconstruction of the dangling P bonds is likely. However, for the bilayer step, the thin ribbon of (111)*B* surface at the step edge is just large enough to accommodate the unit cells of the (2×2) reconstruction ob-

served by Biegelsen *et al.*[30] on the (111)*B* surface of GaAs. The (2×2) surface is covered by a layer of As trimers bonded to the underlying surface, so it would be expected to form only for group-V-rich conditions. Thus, formation of the reconstructed bilayer structure at the step edge on the GaInP surface would be stabilized only for high phosphine (either PH_3 or TBP) flow rates.

At low V/III ratios the vicinal regions consist mainly of monolayer steps. For either thermodynamic or kinetic reasons, this leads to step bunching. As the V/III ratio increases, the monolayer steps are replaced by predominantly bilayer steps. This changes either the relative energies of the various types of steps on the surface (thermodynamic factor) or the attachment kinetics of adatoms moving ''up'' or ''down'' the step edge (kinetic factor) to eliminate the large supersteps. A similar suppression of step bunching at high V/III ratios has been reported for GaAs layers grown by OMVPE.[31]

It is possible that the mechanism for the effect of V/III ratio on ordering is related to the change in step structure from monolayer to bilayer in the vicinal regions. This may operate independently or in addition to the effect of the change in the surface reconstruction of the (001) facets discussed above. Since these effects all occur in the same range of V/III ratio, the two possible mechanisms are difficult to separate. Interpreted in this way, the data would indicate that bilayer steps assist in the formation of the Cu–Pt structure. The similar dependence of SPA signal and degree of order reported with changing temperature[16] seems to suggest that the predominant factor is the change in surface reconstruction.

The reduction in degree of order observed for V/III ratios higher than the optimum value for both phosphine and TBP appears to be unrelated to the surface reconstruction. Kurtz *et al.*[12] have suggested that this may be due to a postulated reduction in the group-III surface diffusion coefficients. Another possibility is related to changes in the step structure caused by high V/III ratios. For [110]-oriented steps, a group-III atom makes no extra bonds as it approaches the step edge. It makes two bonds on either the unreconstructed or the (2×4) reconstructed surface. However, at a kink site, it may make three bonds if the adjacent group-III atom is covered by a P atom bound to the step by a single bond, a situation likely only for very high V/III ratios. A group-III atom making three bonds at the kink is likely to have a high sticking coefficient. This will give rise to a nearly random distribution of Ga and In atoms as the kink moves along the [110] step edge. In fact, this situation is similar to that occurring at a $[\bar{1}10]$ step edge. The high sticking coefficient due to the three bonds made by the group-III atom at the step is probably responsible for the high growth rate in the $[\bar{1}10]$ direction, the relatively ragged $[\bar{1}10]$ step edges, and the reduction in order observed when $[\bar{1}10]$ steps are produced on the surface due to misorientation from (001) in the [110] direction.[32]

V. CONCLUSIONS

The effects of changing the V/III ratio on ordering have been explored for epitaxial GaInP layers grown using two phosphorus precursors, phosphine and tertiarybutylphosphine. The surface structure was probed using SPA to measure the relative concentration of $[\bar{1}10]$-oriented P dimers on the surface and AFM to characterize the surface steps. The degree of order was observed to decrease markedly with decreasing V/III ratio when the input partial pressure of the phosphorus precursor was reduced below 3 Torr for PH_3 and 0.75 Torr for TBP. The difference between the two precursors is determined to be due mainly to the incomplete pyrolysis of the PH_3. The reduction in order parameter is observed to correlate with a decrease in the SPA signal at 400 nm due to the $[\bar{1}10]$-oriented P dimers on the surface that are characteristic of the (2×4) reconstructed surface. Thus, the reduction in ordering is attributed to a loss of the (2×4) reconstruction for low phosphorus partial pressures. When the partial pressure of the phosphorus precursor is increased beyond the optimum value for ordering, the degree of order decreases gradually. The SPA signal continues to increase in this regime. Thus, the decrease in the degree of order is attributed to an increase in the sticking probability at kinks on the [110] steps for these high V/III ratios. At the lower phosphine (either TBP and PH_3) partial pressures, the [110] steps are observed, using the AFM, to bunch, producing supersteps varying in height from approximately 30 to 40 Å. Above a certain partial pressure, corresponding to that at which ordering is suppressed for both precursors, the step bunching is observed to be nearly eliminated. This corresponds to a change from monolayer to bilayer steps in the vicinal regions which is believed to be related to stabilization of the bilayer steps by formation of the (2×2) reconstruction on the (111)*B* step edge. The width of the step edge for bilayer steps is exactly the size of the (2×2) unit cell and is terminated by P trimers, so would form only for high phosphorus partial pressures.

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