Heterostructures in GaInP grown using a change in V/III ratio

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A natural monolayer {111} superlattice (the CuPt ordered structure) is formed spontaneously during organometallic vapor phase epitaxial (OMVPE) growth of $Ga_{0.52}In_{0.48}P$. The extent of this ordering process is found to be a strong function of the input partial pressure of the phosphorus precursor during growth due to the effect of this parameter on the surface reconstruction and step structure. Thus, heterostructures can be produced by simply changing the flow rate of the P precursor during growth. It is found, by examination of transmission electron microscope (TEM) and atomic force microscope (AFM) images, and the photoluminescence (PL) and PL excitation (PLE) spectra, that order/disorder (O/D) (really less ordered on more ordered) heterostructures formed by decreasing the partial pressure of the P precursor during the OMVPE growth cycle at a temperature of 620 °C are graded over several thousands of Å when PH_3 is the precursor. The ordered structure from the lower layer persists into the upper layer. Similarly, D/O structures produced by increasing the PH_3 flow rate yield PL spectra also indicative of a graded composition at the heterostructure. The grading is not reduced by a 1 h interruption in the growth cycle at the interface. Similar heterostructures produced at 670 °C using tertiarybutylphosphine (TBP) as the P precursor show a totally different behavior. Abrupt D/O and O/D heterostructures can be produced by abruptly changing the TBP flow rate during the growth cycle. PL and PLE studies show distinct peaks closely corresponding to those observed for the corresponding single layers. TEM dark field images also indicate that the interfaces in both for D/O and O/D heterostructures are abrupt. The cause of the difference in behavior for TBP and PH_3 is not clear. It may be related to the difference in temperature. © *1997 American Institute of Physics.* [S0021-8979(97)01311-X]

I. INTRODUCTION

Atomic-scale ordering, i.e., the spontaneous segregation of atoms in a ternary alloy into alternating crystallographic planes, is a phenomenon observed in many semiconductors including essentially all III/V alloys.[1] Most commonly for III/V alloys, the atoms segregate to produce an atomic-scale modulation on one of the {111} planes, forming the so-called CuPt structure. This phenomenon is of fundamental interest in terms of the thermodynamics of III/V alloys and the surface processes occurring during organometallic vapor phase epitaxial (OMVPE) growth. It is also of practical interest because ordering has a significant effect on the materials properties, e.g., the band gap energy is found to be 160 meV less in partially ordered $Ga_{0.52}In_{0.48}P$ than in disordered material of the same composition.[2] Thus, ordering is carefully avoided in visible light-emitting diodes (LEDs) and injection lasers in order to produce the highest emission energies.[3] On the other hand, ordering offers the possibility of producing heterostructures by changing the band gap energy without altering the solid composition. Such structures might be useful for devices such as lasers, LEDs,[4] and high efficiency solar cells.[5]

Theoretically, the thermodynamic driving force for CuPt ordering is due to formation of rows of $[\bar{1}10]$ oriented P dimers on the (2×4) reconstructed (001) surface.[6] This has been supported by the recent experimental observation, using surface photoabsorption (SPA), that the concentration of $[\bar{1}10]$ P dimers on the (001) surface correlates closely with the degree of order produced during OMVPE growth: Both the SPA signal and the measured degree of order(S) are observed to decrease with increasing temperature[7,8] and decreasing phosphorus precursor (tertiarybutylphosphine, TBP) flow rate[8–10] within certain ranges.

An intriguing correlation between the degree of order and the step structure on the surface has also been observed.[8,11,12] Atomic force microscopic (AFM) examination of the surfaces of GaInP epitaxial layers grown on singular (001) GaAs substrates reveals a mixture of monolayer and bilayer steps. The ratio of monolayer to bilayer steps for layers grown using TBP depends strongly on the growth parameters. Both the degree of order and the fraction of bilayer steps are seen to increase monotonically with increasing partial pressure of the P precursor[8,11] and increasing temperature.[11] An increase in growth rate with a constant TBP flow rate was found to have no effect on either the degree of order or the step structure of the GaInP grown on singular (001) GaAs substrates.[12] Interpretation of the data is

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complicated by the simultaneous change in the concentration of $[\bar{1}10]$ surface dimers and the step structure with changing temperature and TBP partial pressure.

Heterostructures[13–15] and quantum wells[16] can be produced using the abrupt change in order parameter induced by a change in the growth temperature, with absolutely no change in the solid composition. This avoids problems such as lattice mismatch at the interface. The production of both single and double heterostructures[13–15] as well as quantum wells with ordered layer wells with nominal thicknesses as small as 10 Å[16] have been produced in this way. The changes in degree of order and band gap energy are found to be abrupt using this technique.

However, as a practical process, using a change in temperature to produce the desired change in order parameter is inconvenient. At each interface the growth must be interrupted for several minutes to allow the temperature to be adjusted and stabilized.[13–16] This may, of course, result in

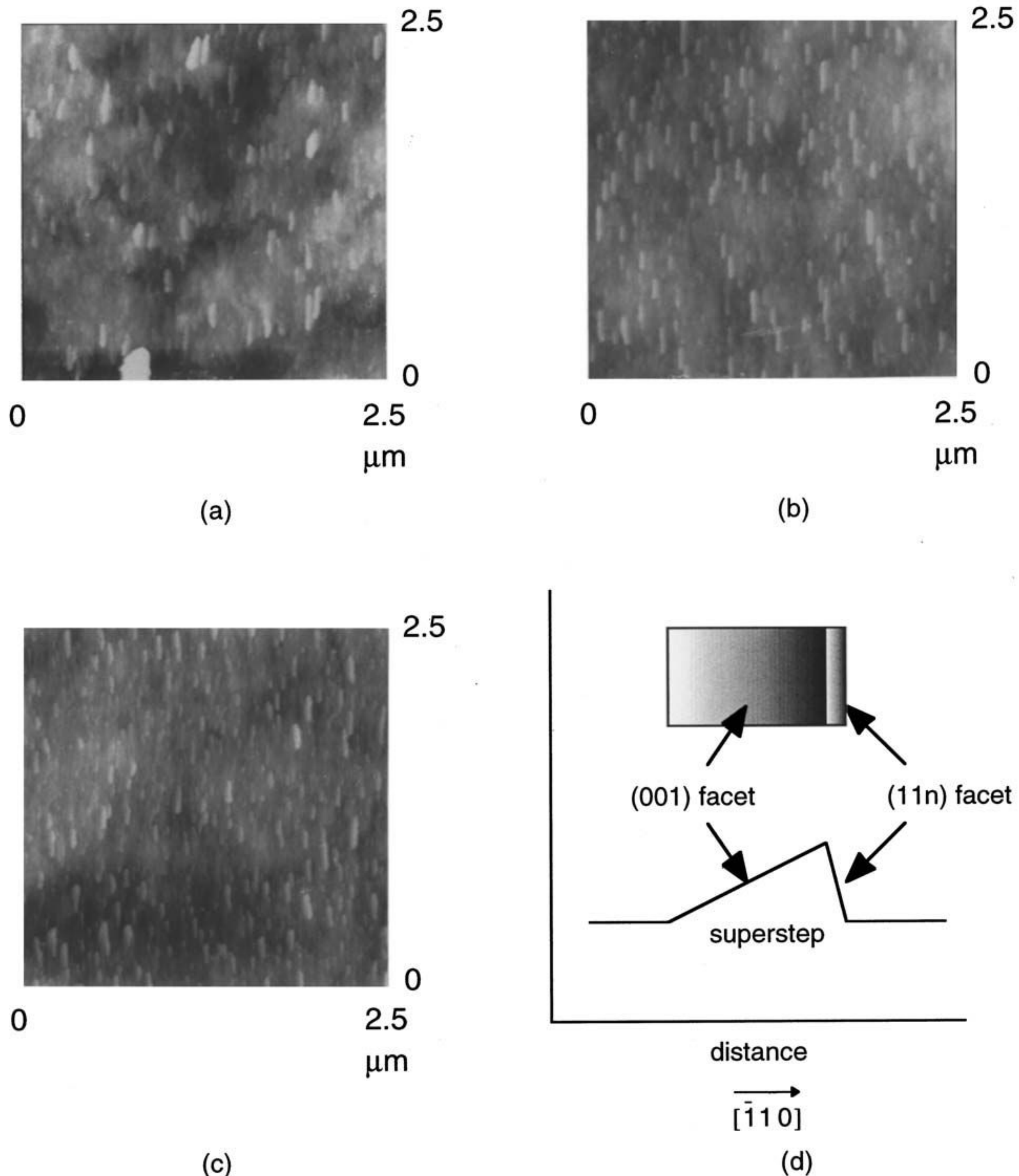

FIG. 1. AFM images of individual $Ga_{0.52}In_{0.48}P$ layers and heterostructure grown on 3° misoriented substrates (toward $\{111\}_B$) at a temperature of 620 °C using V/III ratios of 40 (a), 160 (b), and 160/40 (c) using PH_3 as the P precursor; schematic diagram of the superstep (d).

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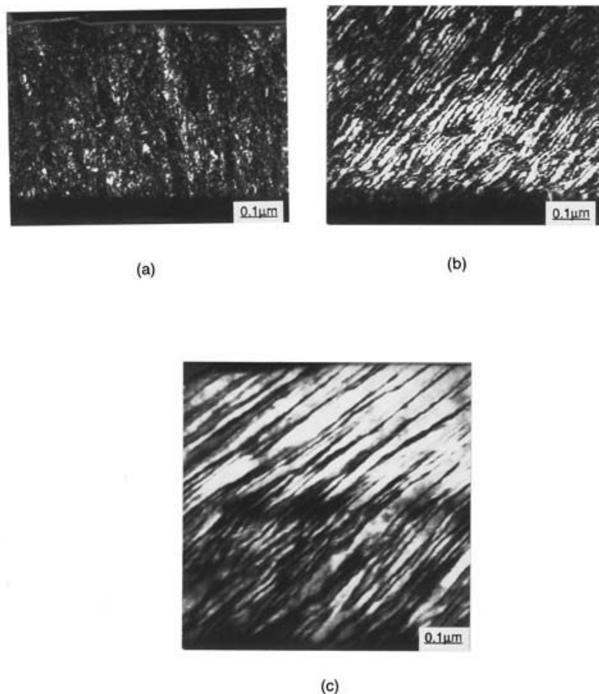

FIG. 2. TEM images of individual $Ga_{0.52}In_{0.48}P$ layers and heterostructure shown in Fig. 1 using V/III ratios of 40 (a), 160 (b), and 160/40 (c) using PH_3 as the P precursor.

contamination of the interface. In addition the group III flow rates should be adjusted during the interruption since a change in temperature results in a slight change in solid composition. In principle, a much more attractive approach would be to change the flow rate of the phosphorus precursor to produce the desired change in order parameter, although the changes in band gap energy are typically considerably smaller than for changes in temperature.[8–10]

This paper is concerned with the results obtained using a change of flow rate of the P precursor, either phosphine or TBP, to produce heterostructures in GaInP. For PH_3, this procedure is used to produce order/disorder (O/D) (less ordered on more ordered) heterostructures consisting of a less ordered(D) layer with low PH_3 flow rate grown upon a more ordered(O) first layer grown using a high PH_3 flow rate at 620 °C. In practice, the ordering in the bottom layer is found to persist for thousands of Å into the second layer. Similar D/O(more ordered on less ordered) structures are also not abrupt. However, using TBP at 670 °C an entirely different result is obtained. Both O/D and D/O structures are produced using TBP, giving photoluminescence excitation (PLE) peaks from the two individual layers and with abrupt interfaces.

II. EXPERIMENT

The GaInP epitaxial layers were grown by OMVPE on (001) semi-insulating GaAs substrates, either singular or misoriented by an angle of 3° toward the $[\bar{1}10]$ direction or more concisely the *B* direction. Substrate preparation consisted of standard degreasing followed by a 1 min etch in a solution of $12H_2O{:}2NH_4OH{:}1H_2O_2$. The substrates were then rinsed in de-ionized water for 5 min and blown dry with N_2 before loading into the reactor. The growth was carried out in a horizontal, atmospheric pressure reactor using trimethylgallium (TMGa) and trimethylindium (TMIn) held in baths at temperatures of -7 and 25 °C, respectively, with Pd-diffused hydrogen as the carrier gas. The phosphorus sources were either phosphine or TBP. The growth temperature was either 620 or 670 °C. The input partial pressure of the phosphorus precursor was varied with constant group III flow rates to produce the heterostructure. The growth rate was constant at 0.5 μm/h, corresponding to 1/2 monolayer/s. Before beginning the GaInP growth, a 0.15 μm GaAs buffer layer was deposited using TMGa and arsine to improve the quality of the GaInP layers.

The solid composition of the GaInP layers was measured by x-ray diffraction using Cu K_α radiation. Only results for nearly lattice matched layers are presented. The photoluminescence (PL) was excited either with the 488 or the 514 nm line from an argon ion laser. The PLE was excited with 615–680 nm light from a Coherent CR-599 dye laser with DCM dye and detected using conventional techniques. [110] cross-sectional transmission electron microscope (TEM) samples were prepared using standard Ar-ion milling at 77 K. The transmission electron diffraction (TED) patterns and TEM images were obtained using either a JEOL 2000 FXII or JEM 2010 scanning TEM operated at 200 kV. A SPA system attached to the OMVPE system using TBP 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. 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.

The surface structure was characterized using a Nanoscope III 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/s were used and data were taken at 12 points/line and 512 lines/scan area.

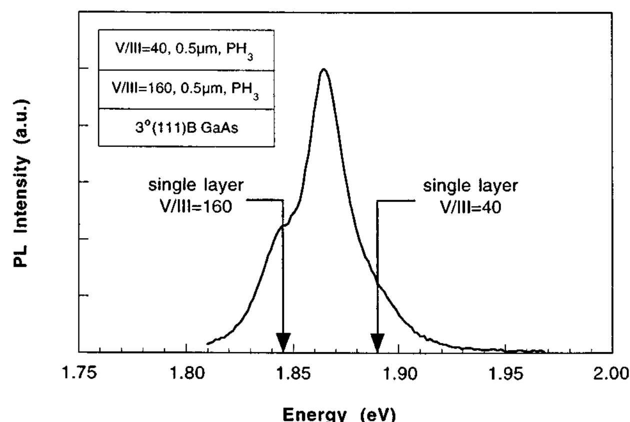

FIG. 3. Schematic diagram and 15 K PL spectrum from the order/disorder heterostructure shown in Fig. 2. The peak positions observed for the individual layers grown using these conditions are also indicated.

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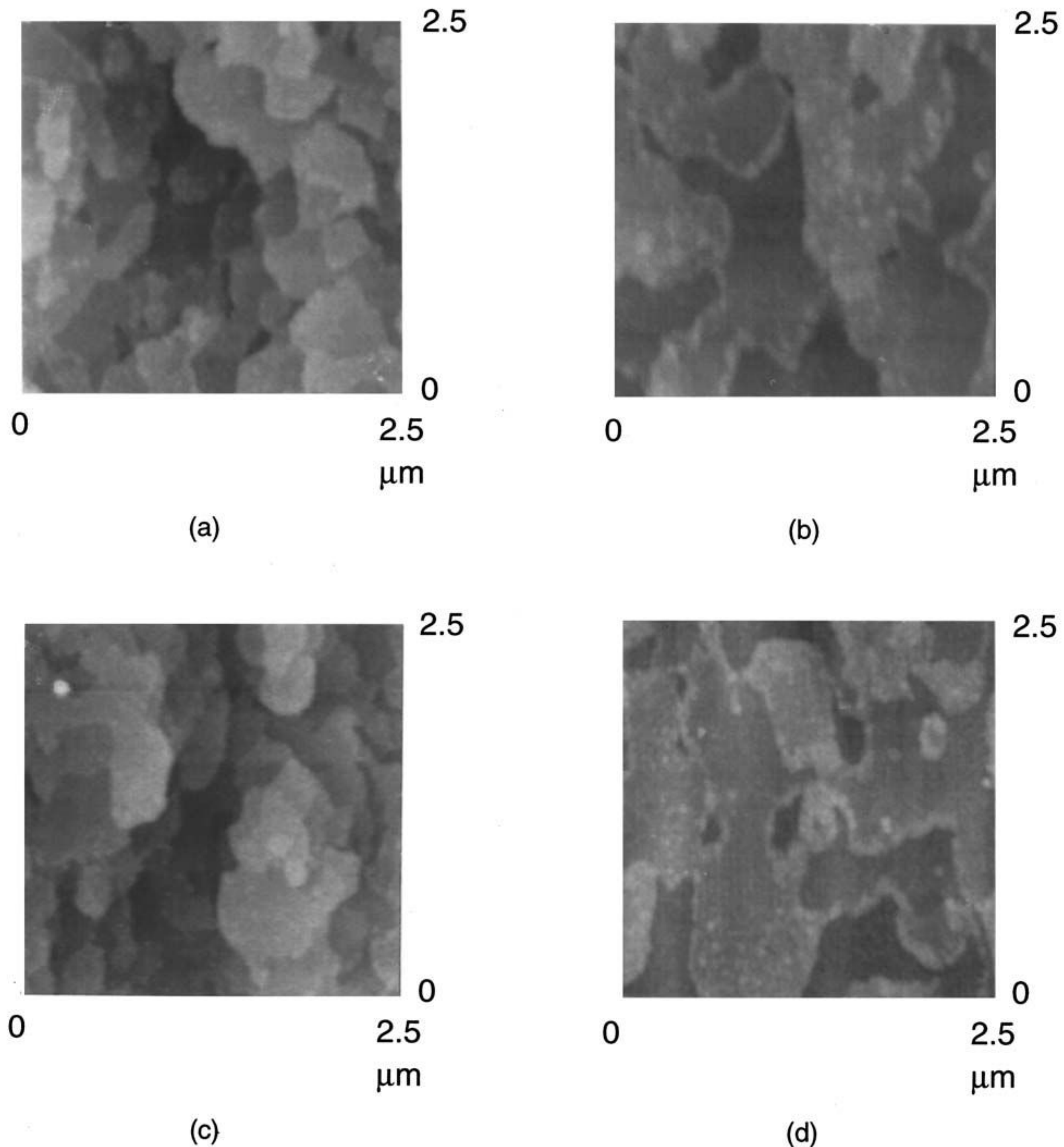

FIG. 4. AFM images of individual $Ga_{0.52}In_{0.48}P$ layers and heterostructure grown on a singular (001) substrate at 670 °C using V/III ratios of 40 (a), 160 (b), 40/160 (c), and 160/40 (d) using TBP as the P precursor.

III. RESULTS

A. Growth of heterostructures using phosphine

Single and double heterostructures, using temperature to modulate the order parameter, were previously successfully grown using PH_3 at a temperature of 620 °C for the ordered layer and 740 °C for the disordered layer on substrates misoriented by 3° in the *B* direction.[13–15] A temperature of 620 °C was used for the first experiments described here with a change in the PH_3 flow rate from 160 to 40 used to produce an order/disorder heterostructure while keeping the flow rates of the TMGa and TMIn constant.

The AFM images shown in Figs. 1(a) and 1(b) are for single layers of GaInP grown on substrates misoriented by 3° in the *B* direction with V/III ratios of 40 and 160. Step bunching (superstep formation) occurs, producing surfaces consisting of 3 ''phases,'' (001) facets, (11*n*) facets, and relatively large vicinal regions in between. The schematic diagram of a superstep is also shown in Fig. 1(d). The spacing of the supersteps produced by step bunching decreases as the V/III ratio increases from 40 to 160. This is consistent with results of an earlier systematic study.[10] Figure 1(c) shows the AFM image for a single heterostructure produced

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at 620 °C by growing the first layer using a V/III ratio of 160 and then changing the PH_3 flow rate to give a V/III ratio of 40. The AFM image of the top layer grown with a V/III ratio of 40 is very different from that of the single layer grown with this V/III ratio: It is similar to the morphology of the single layer grown with a V/III ratio of 160.

Figure 2 shows the TEM dark field cross sections for the layers seen in Fig. 1. The layer grown with a V/III ratio of 40, seen in Fig. 2(a), consists of small domains of ordered material. The 15 K PL peak energy is determined to be 1.89 eV. The layer grown with a V/III ratio of 160 has a structure typical of highly ordered layers, with antiphase boundaries (APBs) clearly observed and large domains extending from the substrate through the entire layer, as seen in Fig. 2(b). The 15 K PL peak from this sample is observed at an energy of 1.845 eV. The dark field TEM image for the O/D heterostructure is shown in Fig. 2(c). The TEM image of the bottom layer closely resembles that seen in Fig. 2(b) for the single layer grown using the same conditions. Surprisingly, the TEM image of the top layer differs greatly from that seen in Fig. 2(b): The layer appears to be much more highly ordered, with larger domains. In fact, it appears that the ordered structure from the bottom layer propagates into the top layer. TED patterns also show approximately the same superlattice spot intensities for both layers in the heterostructure.

The 15 K PL spectrum and schematic diagram for this heterostructure are shown in Fig. 3. The PL is dominated by a single broad peak, with an energy approximately midway between those measured for the individual layers grown at V/III ratios of 40 and 160, which are indicated in Fig. 3. A well-defined low energy shoulder is observed at approximately the position expected for the layer grown at a V/III ratio of 160. There may also be a much weaker shoulder on the high energy side at 1.89 eV, the value measured for the single layer grown at a V/III ratio of 40. This PL spectrum is indicative of a gradual increase in band gap energy throughout the top layer. The PL results appear to be consistent with the TEM and AFM images. The first layer, grown at a V/III ratio of 160, appears to be similar in the TEM image and the PL spectrum to the single layer grown using the same conditions. However, the ordering in the second layer changes only slowly when the PH_3 flow rate is decreased. This results in a transition layer which apparently gives the main PL peak seen in Fig. 3. Interruptions as long as 1 h at the interface caused no significant improvements in the PL results. Apparently the degree of order does eventually (at a distance of less than 0.5 μm from the interface) approach that observed for a single layer grown at a V/III ratio of 40. However, the microstructure of the cross section and step structures on the surface appear to persist even to the top surface. Similar results were obtained for heterostructures grown using PH_3 at 620 °C on singular (001) substrates, although the surfaces are too rough to allow clear AFM images of the steps. The 15 K PL consists of a broad single peak at a position between those expected for single layers grown at V/III ratios of 160 and 40. Thus, the ''memory'' effect seems to be independent of substrate misorientation.

B. Growth of heterostructures using TBP

Both the O/D and D/O types of heterostructures were grown at 670 °C using TBP on substrates that were singular or misoriented by 3° in the *B* direction. For a singular (001) substrate, the D/O heterostructures were produced by first growing a layer with a V/III ratio of 40 followed by the growth of a second layer using a V/III ratio of 160. O/D structures were grown using similar conditions with the order of the layers reversed. Figure 4 shows the AFM images for the single GaInP layers and the heterostructure grown on singular substrates. The surface of the GaInP layer grown at a V/III ratio of 160(b) is seen to consist of large, easily distinguished islands. For the sample grown at a V/III ratio of 40(a) the islands are markedly smaller. AFM examination of the surfaces of the single layers shows a clear increase in the fraction of steps that are bilayers (bilayer/monolayer+bilayer) from approximately 0.3 for a V/III ratio of 40 to 0.7 for a V/III ratio of 160. For both O/D and D/O heterostructures, the top layers have the same fraction of bilayer steps as the single layers. These results are completely different from those obtained using PH_3.

This experiment was repeated using a substrate misoriented by 3° in the *B* direction. As seen in Fig. 5, AFM images indicate that the step structure of the top layer is virtually the same as for the single layer grown using the same conditions for both cases. The PL spectrum from the D/O structure is shown in Fig. 6(a). It is a combination of several peaks. They cannot be unambiguously shown to be the sum of the PL peaks from the single layers. To identify which PL peaks come from which layers and to unambiguously determine the difference in band gap energy between the two layers PLE spectra from the D/O heterostructure are also shown in Fig. 6. The PL peaks labeled *b* and *c* are identified as coming from the less ordered (D) and more ordered (O) layers, respectively. The PLE spectrum shown in Fig. 6(b), and detected on the peak labeled *b* in Fig. 6(a), shows that this emission is due to excitons formed in the more ordered layer. The peak in the PLE spectrum is due to the free excitonic absorption and is very close in energy to a similar feature in the PLE spectrum of a nominally equivalent single-layer sample. The emission peak labeled *c* is identified as coming from the more ordered layer through an analysis of its PLE spectrum, shown in Fig. 6(c). In Fig. 6(c), the PLE data are shown as open circles. The *triangles* are a weighted sum of a PLE spectra taken from the more ordered single layer and the less ordered single layer grown using the same conditions. The qualitative agreement between the data and this sum supports our model that this emission comes from the more ordered layer. The light is absorbed in the less ordered, higher band gap layer followed by movement of the minority carriers into the more ordered, lower band gap layer where they recombined.[17] The PLE spectra show a peak separation of approximately 25 meV in both heterostructures. The PLE results clearly show that the two layers in both for D/O and O/D heterostructures have the same band gaps as the individual layers grown singly. The absence of

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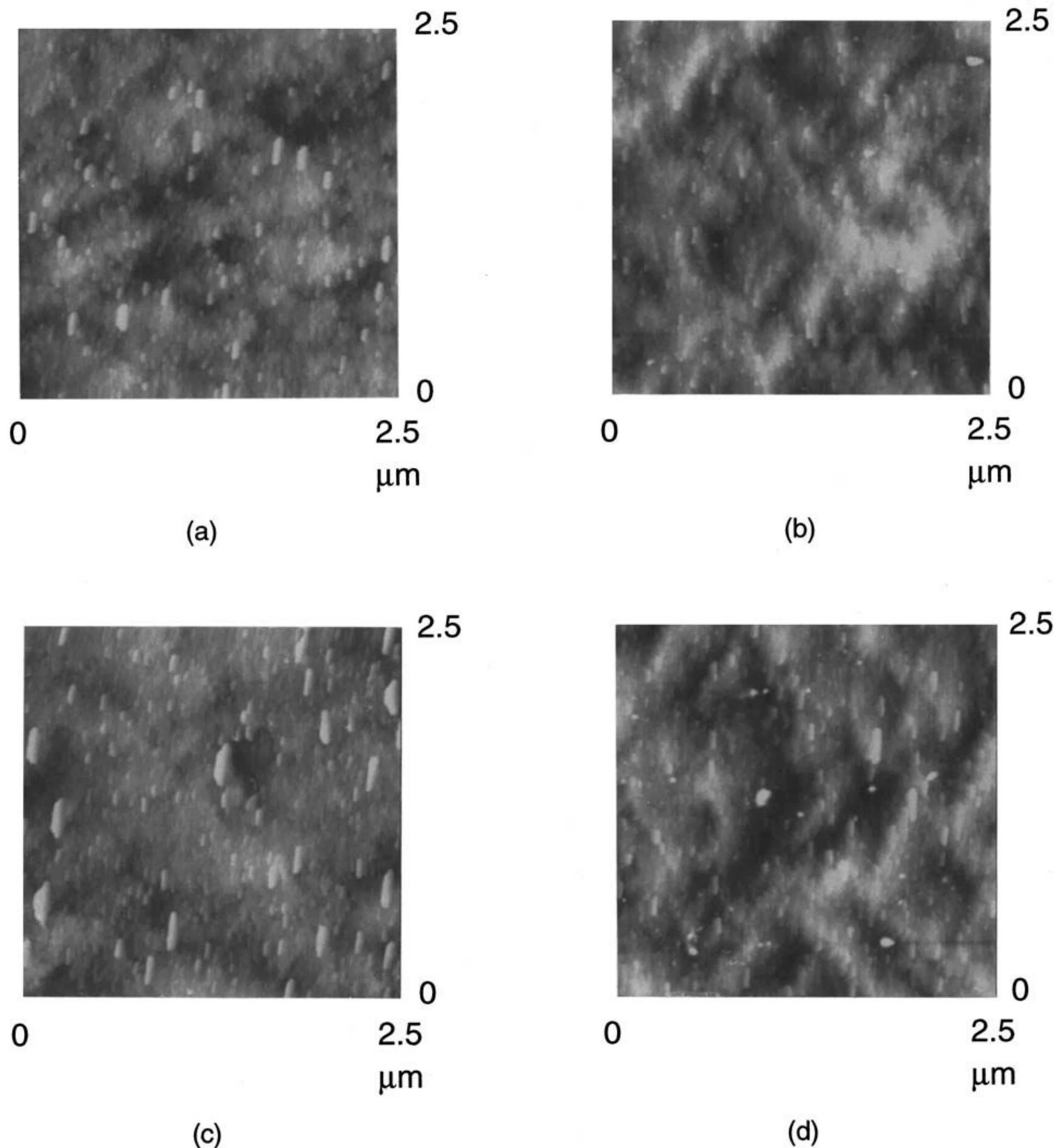

FIG. 5. AFM images of individual $Ga_{0.52}In_{0.48}P$ layers and heterostructure grown on 3° misoriented substrate in *B* direction at a temperature of 670 °C using V/III ratios of 40 (a), 160 (b), 40/160 (c), and 160/40 (d) using TBP as the P precursor.

PL peaks that cannot be accounted for from the individual layers indicates the absence of the graded layers that dominated the PL spectrum of similar structures grown using PH_3.

[110] pole TED patterns for the D/O and O/D heterostructures grown on substrates misoriented by 3° in the *B* direction are shown in Fig. 7. The TED patterns show order-induced superspots due mostly to a single variant for both samples. This is consistent with previous reports for a substrate misorientation of 3° where the ordered regions were found to be essentially all of the same variant.[18,19] The substrate misorientation has reduced the intensity of one variant significantly. Since the domains are large, the superspot intensities give a good indication of the degree of order.[18] It is clearly higher for the layer grown with a V/III ratio of 160 in both for O/D and D/O heterostructures. The TEM dark field images indicate that the interfaces are abrupt. This result is consistent with the PLE results. The data make it clear that abrupt heterostructures can be produced by changing the TBP partial pressure during growth at 670 °C. There is no sign of the grading observed for the heterostructures grown using PH_3 at 620 °C.

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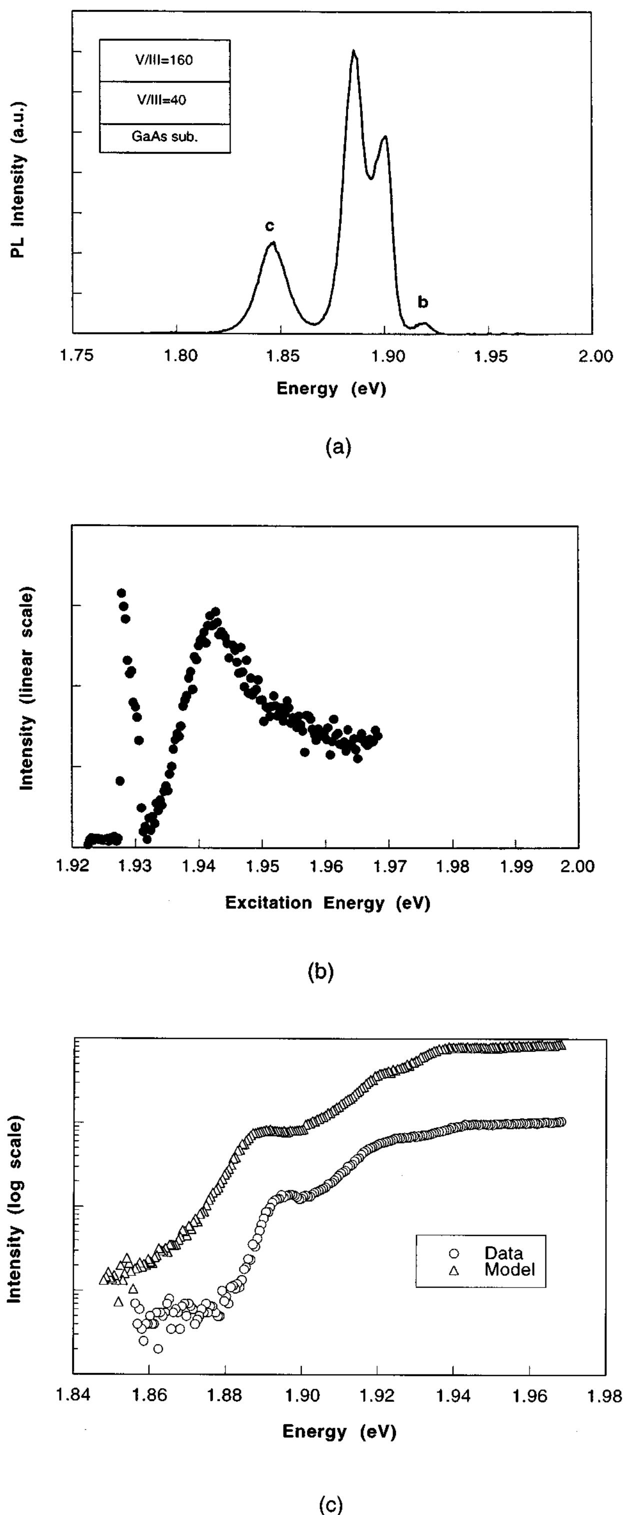

FIG. 6. The 5 K PL spectrum and PLE spectra for disorder/order heterostructure grown on an exactly (001) substrate at 670 °C using TBP by changing the V/III ratio from 40 to 160 during growth. (a) PL spectrum, (b) PLE for PL peak *b* (c) PLE for PL peak *c*.

IV. DISCUSSION

Both O/D and D/O heterostructures have been successfully grown using TBP as the phosphorus source at 670 °C. This is true for substrates having the singular (001) orientation as well as those misoriented in the *B* direction by an angle of 3°. Band gap energy separations as large as 25 meV are obtained from the PLE results. This contrasts sharply with the results obtained using PH_3 as the P precursor at 620 °C, where the heterostructure was graded over thousands of Å.

Formation of the CuPt ordered structure is expected, and experimentally observed, to be driven by formation of the (2×4) reconstructed surface.[6–10] Thus, one might expect the dynamics of the change in order parameter to mimic the change in the surface reconstruction. We investigated the time dependence of the SPA signal at 405 nm, due to $[\bar{1}10]$ P dimers, when the partial pressure of TBP was changed from 10 to 200 Pa at a temperature of 670 °C. The transition time is equal to the measured response time of the mass flow controller of approximately 10 s. Data for a decrease in the TBP partial pressure from 200 to 10 Pa show a similar transition, but the rate is somewhat slower due to the slower mass flow controller response rate (40 s). This time corresponds to a thickness of 20 monolayers ($\sim$30 Å). These data show conclusively that the change in the reconstruction of the surface is virtually instantaneous at 670 °C when TBP is the precursor. Since the step structure observed using AFM is also rapid, an abrupt heterostructure results from a change in the TBP flow rate.

In contrast, both the O/D and D/O heterostructures produced using PH_3 are graded over thousands of Å. This is true for growth at temperatures of 620 °C for singular (001) substrates as well as those misoriented by an angle of 3° in the *B* direction. The reasons for the graded heterostructure obtained using PH_3 are not clear at present. Unfortunately, the SPA apparatus is not configured for the use of PH_3, so neither the reconstruction nor the transient in the SPA spectra when the PH_3 flow rate is changed have been measured.

One possible reason for the graded heterostructure is suggested by the AFM results. The step structure from the bottom layer persists long after the PH_3 flow rate is changed. This does not occur for the TBP. Our previous results[11,12] suggested a link between the step structure and ordering, although the relationship is blurred by the simultaneous change in the surface reconstruction and the step structure as the P partial pressure is varied. Nevertheless, it is possible that the sluggish change in order parameter is linked to the slow change in step structure as the PH_3 partial pressure is changed. The step structure can be observed directly using the AFM. Individual steps can be observed on the surface for singular (001) surfaces.[18] For low V/III ratios the steps on singular (001) surfaces are found to be predominately 2.8 Å in height or monolayer steps for both TBP and PH_3. At high V/III ratios, the (001) surface is seen to consist mainly of bilayer steps. This appears to be the same for the vicinal regions using misoriented substrates, although the data are less reliable due to the high step density. The change in step structure on the surface produced by changing PH_3 flow rate at 620 °C seems to be much slower than for growth using TBP at 670 °C since the step structure and the microstructure of the top layer are the same as those of the bottom layer when PH_3 is used as the P precursor. This slow change in step structure may possibly account for the lag in change in order parameter after the V/III ratio is changed. Since the

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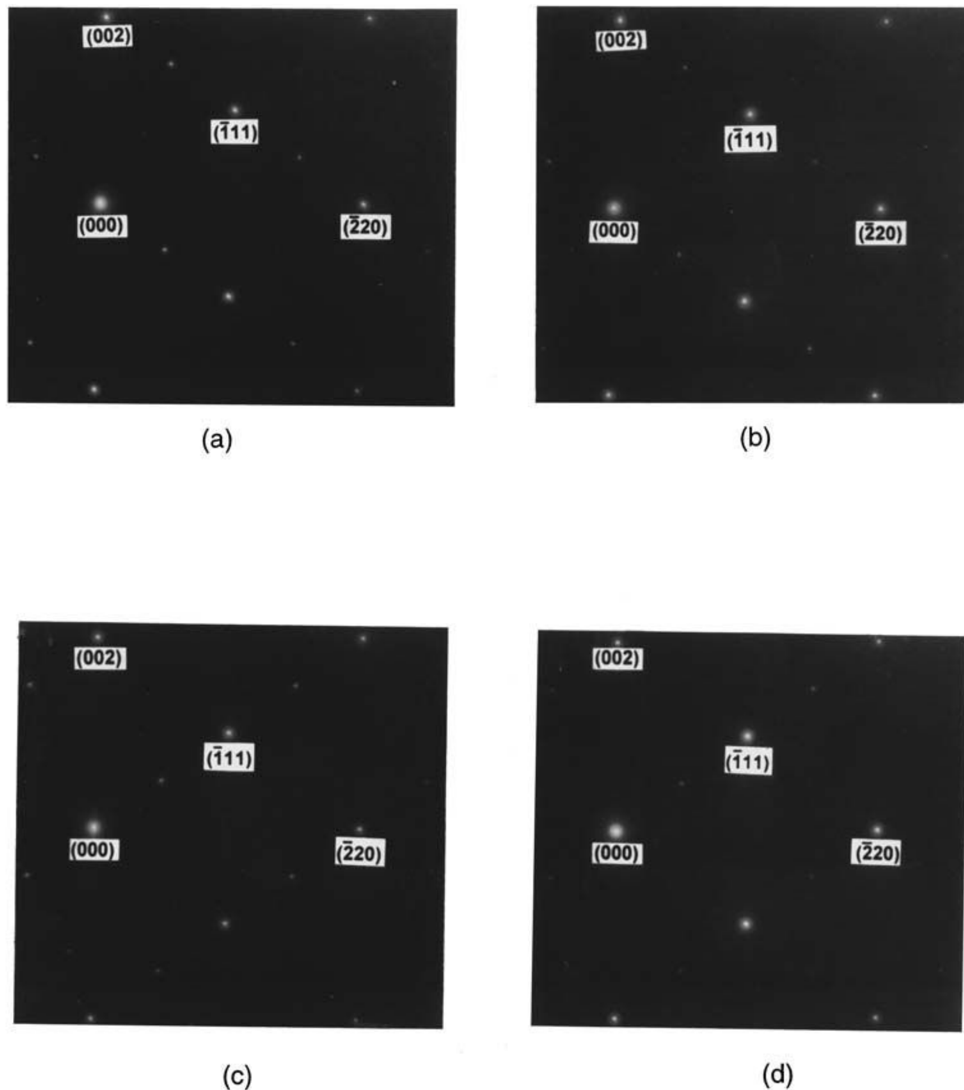

FIG. 7. [110] transmission-electron diffraction patterns obtained for GaInP heterostructures grown at 670 °C on GaAs substrates misoriented by 3° in the *B* direction using TBP: For D/O heterostructure (a) V/III ratio of 160, (b) 40; for O/D heterostructure (c) V/III ratio of 160, (d) 40.

temperatures were different for the heterostructures grown using TBP and PH_3, further experiments are required to determine for certain whether the effects described are due to the change of temperature or the difference in surface chemistry for the two precursors. The growth process appears to be nominally the same for growth using TBP and phosphine, although TBP is known to pyrolyze at lower temperatures.[20] The GaInP materials properites appear to be nearly independent of the P precursor, both in our laboratory and elsewhere.[21] However, preliminary data indicate that the degree of order obtained for the optimum growth conditions for growth using PH_3 is larger than for TBP at 620 °C.[10]

V. CONCLUSIONS

The degree of CuPt order observed in GaInP layers grown by OMVPE is known to be dependent on the partial pressure of the P precursor during growth. When the V/III ratio was decreased from 160 to 40 for the growth of individual layers at 620 °C using PH_3 on a substrate misoriented from (001) by 3° in the *B* direction, the PL peak energy was found to increase by 45 meV. However, when these two phosphine flow rates were used for the growth of an order/disorder heterostructure, a single broad PL peak was observed covering the entire range of energies of the individual layers. Electron microscope images show that the order from the bottom layer persists for thousands of Å into the top layer. Similar PL results were obtained for both order/disorder and disorder/order heterostructures grown using phosphine at 620 °C for singular (001) substrates. Interruptions as long as 1 h at the interface caused no significant improvements in the PL results. Completely dissimilar results were obtained for growth using similar conditions at 670 °C but with TBP as the phosphorus precursor: PLE results are consistent with the formation of more abrupt heterostructures for both order/disorder and disorder/order heterostructures. Similar results were obtained for singular (001) substrates. TEM dark field images and TED patterns indicate that the interfaces in both for D/O and O/D heterostructures are abrupt and the layers have distinctly different order parameters. The reason for the difference in behavior

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for TBP and PH_3 is postulated to be the lag in the change in order parameter caused by a slow change in the step structure of the surface for growth using phosphine. The change in step structure is much more rapid for growth using TBP. However, the reasons for the difference in behavior for TBP and phosphine are not clear. The effect may also be due to the different temperatures used for growth using the two precursors.

ACKNOWLEDGMENTS

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