

## Step structure and ordering in GaInP

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Steps are known to affect the ordering phenomenon in GaInP;  $[110]$  steps assist ordering and  $[\bar{1}10]$  steps retard ordering. However, the mechanism leading to this behavior has not been determined. In this paper, the step spacing is used as a semiquantitative indicator of the attachment of adatoms at the step edge for organometallic vapor phase epitaxial growth on singular (001) GaAs substrates. The step spacing and degree of order in the epitaxial layers have been studied as a function of temperature and the flow rate of the P precursor for both tertiarybutylphosphine (TBP) and phosphine. As the flow rate of the P precursor is lowered beyond a certain critical point, both the degree of order and the step spacing are seen to decrease together. The behavior is the same for TBP and for  $\text{PH}_3$ . Similarly, as the growth temperature is increased above a certain value, the step spacing and order parameter decrease together. This suggests a relationship between adatom attachment at steps and the ordering mechanism. Possible explanations for these phenomena are explored. At low temperatures, the degree of order is reduced and the step spacing decreases exponentially. The data suggest that the reduction in the surface diffusion coefficient with decreasing temperature is the main factor acting to reduce both the step spacing and the degree of order. However, the increasing adatom sticking coefficient at both step edges expected at low temperatures will affect both phenomena as well. © 1998 American Institute of Physics. [S0021-8979(98)06007-1]

### INTRODUCTION

It was long assumed that the distribution of atoms in semiconductor alloys was random. However, in the last decade it has been discovered that ordering, a phenomenon first observed for metal alloys,<sup>1</sup> also occurs in semiconductors.<sup>2</sup> In ordered alloys, the solid composition is modulated along a particular crystallographic direction with a period of several (most often two) lattice spacings. The most commonly observed structure for III/V alloys, with mixing on either the group III or the group V sublattice, is the CuPt structure, with ordering on  $\{111\}$  planes.<sup>2</sup> This results in formation of natural, monolayer superlattice structures. These materials are no longer cubic, which alters the band structure and so has important consequences for the optical and electrical properties. For example, the band-gap energy of the disordered GaInP lattice matched to GaAs has been observed to shrink by 160 meV in ordered material, even though the GaInP layer is only approximately 50% ordered.<sup>3</sup>

The driving force for formation of the CuPt structure spontaneously during organometallic vapor phase epitaxial (OMVPE) growth is now generally accepted to be due to the formation of a  $(2 \times 4)$ -like reconstruction at the surface.<sup>4</sup> The  $[110]$  rows of P dimers give subsurface strain fields that favor formation of the CuPt structure with ordering on the  $(\bar{1}11)$  and  $(1\bar{1}1)$  planes.<sup>5,6</sup> However, the mechanism by which ordering occurs remains unknown, although a number of speculative models have been developed.<sup>6</sup> In addition to the role of surface reconstruction, it has been shown experimen-

tally that  $[110]$  steps assist the ordering process and  $[\bar{1}10]$  steps retard ordering.<sup>7</sup>

Several experimental observations indicate that kinetic factors are involved in ordering. These probably involve step motion, since this is the last step in the process by which adatoms are incorporated into the lattice. For example, high growth rates lead to disordered material.<sup>8</sup> This is thought to be due to the adatoms having insufficient time to arrange themselves into the lowest free-energy structure at the surface during growth. Obviously, a higher growth rate results in a reduction in the time during which the rearrangement from a disordered into an ordered structure can occur. The disappearance of order at low temperatures<sup>9,10</sup> may be related to the same phenomenon, but in this case the reduction in order would be due to the reduced adatom diffusion coefficient on the surface.

The study of the kinetic processes occurring on the surface and at the step edge during epitaxial growth is worthwhile, since control of the ordering process may be technologically useful for a number of devices. For example, heterostructures can be produced with a change of band gap of 160 meV by simply controlling the degree of order in two layers having exactly the same composition.<sup>3</sup>

In this paper, the step spacing, determined using atomic force microscopy (AFM), was used to monitor processes occurring on the surface and at the step edges. The step spacing and degree of order were examined as a function of the key growth parameters temperature and partial pressure of the P precursor [for both tertiarybutylphosphine (TBP) and  $\text{PH}_3$ ]. Surprising similarities in the step spacing and order parameter were observed.

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## EXPERIMENT

The GaInP layers described in this study were grown by OMVPE in a horizontal, infrared-heated, atmospheric pressure reactor using trimethylindium (TMIn), trimethylgallium (TMGa), and either TBP or  $\text{PH}_3$  on singular (001) semi-insulating GaAs substrates. The carrier gas was Pd-diffused hydrogen. The input partial pressure of the P precursor was varied with a constant group III flow rate to vary the V/III ratio. The growth temperature was varied from 570 to 720 °C. The growth rate was 0.5  $\mu\text{m}/\text{h}$  and layer thicknesses were 0.25  $\mu\text{m}$  for all layers. Substrate preparation consisted of degreasing followed by a 1 min etch in a  $2\text{HN}_4\text{OH}:12\text{H}_2\text{O}:1\text{H}_2\text{O}_2$  solution. Before beginning the GaInP growth, a 0.15  $\mu\text{m}$  GaAs buffer layer was deposited to improve the quality of the GaInP layer. The substrates were heated to the growth temperature in  $\text{AsH}_3$ . After the growth cycle was completed, the samples were cooled with an initial cooling rate of approximately 70 °C/min.

The solid composition of the GaInP layers was determined using Vegard's law, from x-ray diffraction measurements using Cu  $K\alpha$  radiation. Only results for lattice-matched layers, with values of GaP concentration in the solid of 0.515, are presented here. The 18 K photoluminescence (PL) was excited with the 488 nm line of an  $\text{Ar}^+$  laser. The emission was dispersed using a Spex Model 1870 monochromator and detected using a Hamamatsu R1104 head-on photomultiplier tube. The low-temperature PL peak energy was used to determine the degree of order  $S$  using the equation<sup>11</sup>

$$S = [(2005 - \text{PL peak energy at 20 K (in meV)})/471]^{1/2}.$$

The surface structure was characterized using a Nanoscope III atomic force microscope 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 512 points/line and 512 lines per scan area. The samples were measured in air, so were covered by a thin, conformal oxide layer.

## RESULTS AND DISCUSSION

The surfaces of all of the layers in this study, grown on singular (001) substrates, have a wedding-cake-like morphology composed of islands several atomic layers in height. The edges of the islands are monolayer or bilayer steps with nearly equal spacings.<sup>12,13</sup> Both the wedding-cake morphology and the equal step spacing (step ordering) are indicative of step edge barriers hindering the attachment of adatoms at down steps.<sup>14,15</sup> Simple one-dimensional analysis of the growth process indicates that the step spacing is determined by the surface diffusion coefficient and the kinetic barrier to incorporation at the step edge or, in other words, the adatom sticking coefficient at the step edge.<sup>16</sup> A decrease in either the diffusion coefficient or the sticking coefficient at the step edge will reduce the step spacing.

Figure 1 shows the AFM images for layers grown at 670 °C with increasing  $\text{PH}_3$  partial pressure in the sequence (a) 1.0 Torr, (d) 3.0 Torr, and (b) 6.0 Torr. A clear increase in the island size is seen with increasing  $\text{PH}_3$  pressure. The

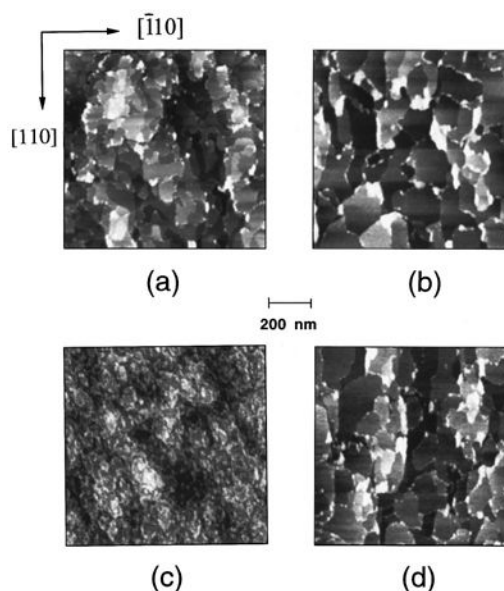


FIG. 1. Atomic force microscopy images of GaInP layers grown at 0.5  $\mu\text{m}/\text{h}$  using  $\text{PH}_3$  as the P precursor. The growth temperature and  $\text{PH}_3$  partial pressure for each run are: (a) 670 °C and 1.0 Torr, (b) 670 °C and 6.0 Torr, (c) 570 °C and 3.0 Torr, and (d) 670 °C and 3.0 Torr.

surface of a sample grown at 570 °C (3.0 Torr of  $\text{PH}_3$ ) is seen in Fig. 1(c). The step spacing is dramatically lower than for the sample grown at 670 °C with the same  $\text{PH}_3$  partial pressure. The average step spacing was obtained from a careful counting along 10 1  $\mu\text{m}$  AFM profiles. It is plotted versus the partial pressure of the P precursor ( $p_P$ ) in Figs. 2(a) and 2(b) for both  $[110]$  and  $[\bar{1}10]$  steps and for the two P precursors TBP and  $\text{PH}_3$ .

Figure 2(a) is for a temperature of 670 °C. Several features of the data are evident. The results for TBP and  $\text{PH}_3$  are virtually identical, consistent with earlier reports,<sup>17</sup> indicating that the pyrolysis is complete for both precursors. This is consistent with surface photoabsorption (SPA) results showing the same concentration of P dimers on the surface for  $\text{PH}_3$  and TBP at 620 °C.<sup>18</sup>

The step spacing for both types of steps at 670 °C is nearly constant at high values of  $p_P$  and drops precipitously as  $p_P$  is decreased below a critical value of  $p_P^*$  of approximately 3 Torr. By 1 Torr the step spacings have decreased by more than a factor of 2. The  $[\bar{1}10]$  step spacing is greater than for the  $[110]$  steps. The difference is larger at the high partial pressures where step spacing is nearly independent of  $p_P$ . At the lower partial pressures, the results are more nearly equal.

The data in Fig. 2(b) are for a growth temperature of 620 °C. The step spacings are smaller, particularly at the high partial pressures, and, as for 670 °C, are always larger for  $[\bar{1}10]$  than for  $[110]$  steps. The temperature dependence will be discussed below. Again, the step spacing is constant at high values of  $p_P$  and decreases below the critical value of approximately 1–1.5 Torr. The value of  $p_P^*$  is lower at 620 than at 670 °C.

In some ways, the data are similar to the results obtained on larger, photolithographically produced, GaAs islands

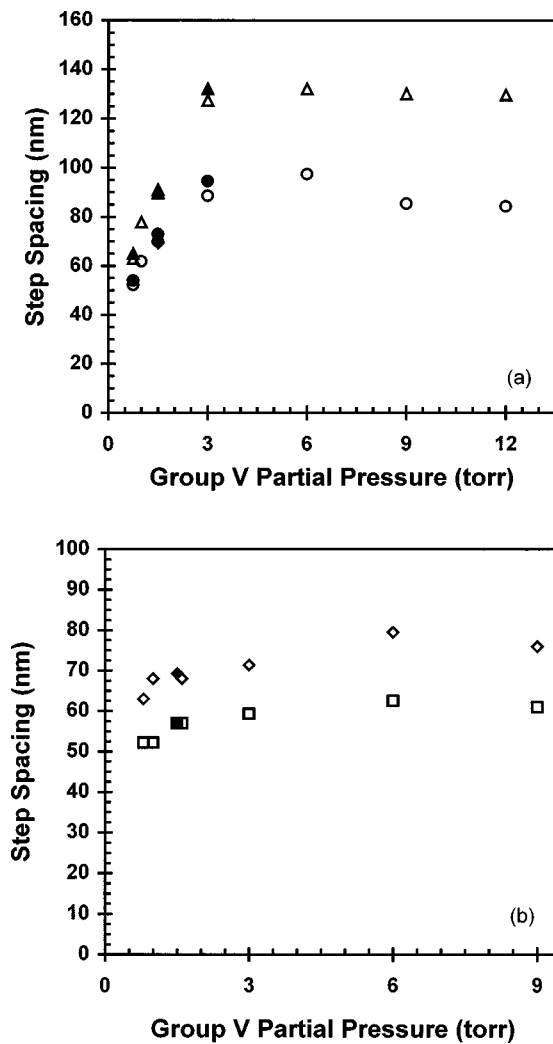


FIG. 2. Step spacing vs the partial pressure of the group V precursor: (a) 670 °C for [110] ( $\Delta$ ,  $\blacktriangle$ ) and [110] ( $\circ$ ,  $\bullet$ ) steps. (b) 620 °C for [110] ( $\diamond$ ,  $\blacklozenge$ ) and [110] ( $\square$ ,  $\blacksquare$ ) steps. The P precursor was either TBP (filled data points) or PH<sub>3</sub> (open data points).

grown by OMVPE on (001) substrates by Asai.<sup>19</sup> (Note the notation used for the step directions is reversed in this paper.) At high values of AsH<sub>3</sub> partial pressure, Asai found the lateral growth rate in the [110] direction to be about twice the [110] growth rate. Both were constant at high group V partial pressures. As the AsH<sub>3</sub> pressure was decreased, the [110] step spacing was observed to drop, while the [110] step spacing was constant. This contrasts with the present data, where the step propagation velocities, or step spacings, decrease together as the P partial pressure is decreased. Asai explained his results in terms of the atomic configuration at the step edge. At the [110] step at high group V partial pressures where the singly bonded group V atom sites at the step edge are saturated, a group III adatom is able to make three bonds. As the group V partial pressure decreases, the probability of finding these singly bonded As atoms decreases, resulting in a decrease in the sticking coefficient of the group III adatoms at the [110] step edge. Of course, this explanation ignores reconstruction of both the surface and the step edge, so it must be viewed as merely schematic. The actual step structure has not been determined. However, step recon-

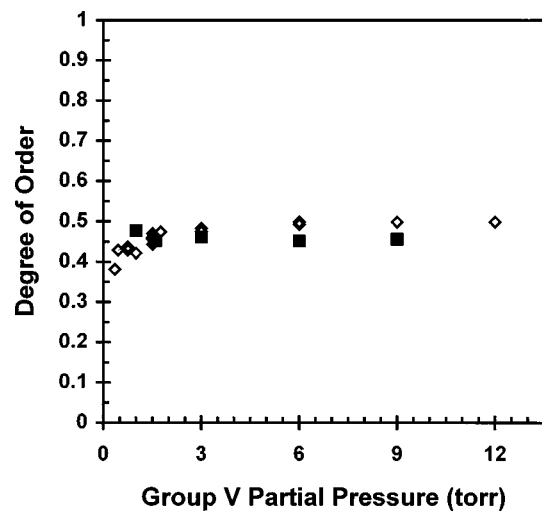


FIG. 3. The degree of order from PL peak energy vs the partial pressure of the group V precursor, either TBP or PH<sub>3</sub>, for growth at 670 ( $\diamond$ ) and 620 °C ( $\blacksquare$ ).

struction is believed to occur in Si,<sup>20</sup> and simple bonding considerations indicate that it will probably also occur in III/V semiconductors as well.

At the [110] step edge, the group III adatom is able to make only two bonds, the same as on the flat (001) terraces. This was the explanation for Asai's observed independence of the [110] lateral growth rate on the group V partial pressure. For the results presented here, the drop in [110] step spacing at low values of  $p_P$  may be related to the sticking coefficient at the *kinks*. In this case, the kinks have an overhanging, singly bonded group V atom site that would be occupied at high group V partial pressures and vacant at low group V partial pressures. If sticking at the [110] step is postulated to occur at kink sites, this would explain the decrease in [110] step spacing as the value of  $p_P$  is decreased. The magnitude of the step spacing is much less than the expected adatom diffusion length, indicating that the sticking coefficient is small for all the growth conditions studied.<sup>21</sup>

The difference between the values of  $p_P^*$  at the two temperatures can be naturally explained using the same argument. At 670 °C a higher value of  $p_P$  is required to assure saturation of the singly bonded P sites than at 620 °C where the P vapor pressure over GaInP is lower.

In Fig. 3, the degree of order, determined from the PL peak energy, is plotted versus  $p_P$  for the same samples used to obtain the data in Fig. 2. The results for TBP and PH<sub>3</sub> are, again, nearly identical. The dependence of the order parameter on the P partial pressure is remarkably similar to that of the step spacing seen in Figs. 2(a) and 2(b). At 670 °C the degree of order begins to drop as the value of  $p_P$  becomes less than approximately 2–3 Torr. At 620 °C, this critical value of  $p_P$  is <1 Torr, again, lower than at 670 °C. The decrease in order parameter at low values of P partial pressure has been observed previously.<sup>11,22</sup> It has been clearly shown to correlate with the surface photoabsorption signal due to [110]-oriented P dimers on the (001) surface.<sup>11</sup> The SPA and ordering data suggest that above the critical value of  $p_P$  a single (2×4) phase is formed that gives a large

driving force for formation of the CuPt structure. The SPA signal is found to be strong for growth under these conditions.<sup>23</sup> The system is thermodynamically invariant and the degree of order is independent of  $p_P$ . Although the data presented here indicate a constant degree of order at high values of  $p_P$ , several earlier studies have indicated a slight decrease in the degree of order at high flow rates of the P precursor.<sup>24,25</sup> This has been attributed to various factors, including the formation of a non- $(2 \times 4)$ , "excess phosphorus," reconstruction at high P partial pressures. However, this is somewhat problematic, since it would be inconsistent with the high SPA signal due to  $[\bar{1}10]$  P dimers observed.<sup>23</sup>

At lower values of  $p_P$  the system apparently sequentially changes between the various  $(2 \times 4)$  phases,<sup>26</sup> with each successive phase having a lower coverage of  $[\bar{1}10]$  P dimers, resulting in the sequential reduction in the CuPt order parameter and the SPA signal at 400 nm.<sup>23</sup> This mechanism would suggest that the change in order parameter with  $p_P$  should consist of a series of steps. However, the data are insufficient to allow these to be observed. This interpretation of the data is supported by the theoretical expectation that the driving force for CuPt ordering should decrease as the concentration of the  $[\bar{1}10]$  P dimers decreases.<sup>5</sup>

Consideration of the step spacing and ordering data together presents somewhat of a dilemma. The similarity of the  $p_P$  dependence of the step spacing and the degree of order would seem to indicate that the same mechanism controls both. However, the variation in step spacing is attributed to the atomic arrangement at the step (or kink) while the variation in the degree of order is attributed to the surface reconstruction on the terraces. Naturally, the same trend is expected for both factors. However, the nearly identical values of  $p_P^*$  suggest that the step edge (or kink) structure changes as the surface reconstruction of the terraces between steps changes. For pressures greater than  $p_P^*$  the surface reconstruction, surface diffusion coefficient, and sticking coefficient at the step edge are postulated to be nearly independent of  $p_P$ .

Other interpretations of the data are: (i) It is actually the atomic configuration at the step edge that controls ordering. The concentration of singly bonded P atoms at the  $[\bar{1}10]$  step edge (or the kink on the  $[\bar{1}10]$  step) changes in qualitatively the same way as the concentration of  $[\bar{1}10]$  dimers on the terraces with increasing  $p_P$ , thus explaining the correlation between the degree of order and the SPA data of Murata and co-workers.<sup>11,27</sup> This seems highly unlikely, since an increase in the sticking coefficient at the step edge would be expected to decrease, rather than increase, the degree of order. A sticking coefficient of unity will obviously result in completely disordered material. (ii) The step spacing is really controlled by the surface reconstruction of the terraces between steps. This might be caused by changes in the surface diffusion coefficient as the surface reconstruction is changed, rather than changes in the sticking coefficient. The data are insufficient to either prove or disprove this hypothesis.

The effects of growth temperature on the step spacing are seen in Fig. 4. Data for  $[\bar{1}10]$  and  $[110]$  steps are indicated. Data obtained at 3.0 and 1.5 Torr (for both  $\text{PH}_3$  and TBP) are shown in Figs. 4(a) and 4(b), respectively. Again,

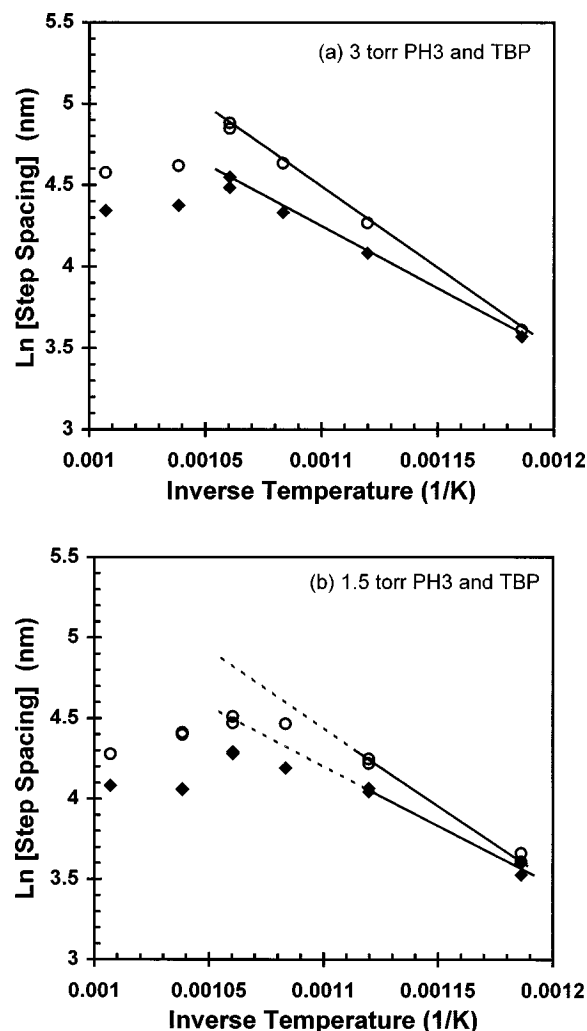


FIG. 4. Log of step spacing vs  $1/T$  for growth using either TBP or  $\text{PH}_3$  with partial pressures of 3 Torr (a) and 1.5 Torr (b).  $[\bar{1}10]$  steps (○) and  $[110]$  steps (◆).

data for  $\text{PH}_3$  and TBP are virtually identical and the  $[\bar{1}10]$  step spacing is typically larger than the  $[110]$  spacing.

The temperature dependence of the data is complex. The largest step spacing occurs for a temperature of approximately 670 °C. At lower temperatures, the step spacing drops exponentially, as indicated by the straight lines on the semi-log plots. The activation energies obtained from Fig. 4(a) are approximately 1.2 eV for the  $[\bar{1}10]$  steps and 1.8 eV for the  $[110]$  steps. The simplest interpretation of the low-temperature behavior is that step spacing is reduced due to the exponential decrease in the surface diffusion coefficient with decreasing temperature. The  $[\bar{1}10]$  step spacing is larger due to the well-known asymmetry in diffusion along and across the  $[\bar{1}10]$  P dimer rows.<sup>16,20</sup> The difference in activation energies is more difficult to explain. One expects diffusion across dimer rows to have a higher activation energy. This problem is probably caused by the fact that the sticking coefficients at the step edges are increasing as the temperature is decreased, even at these low temperatures. The behavior is probably different for the two step directions. Thus, this is probably not a completely reliable method for determining the activation energies for surface diffusion. Never-

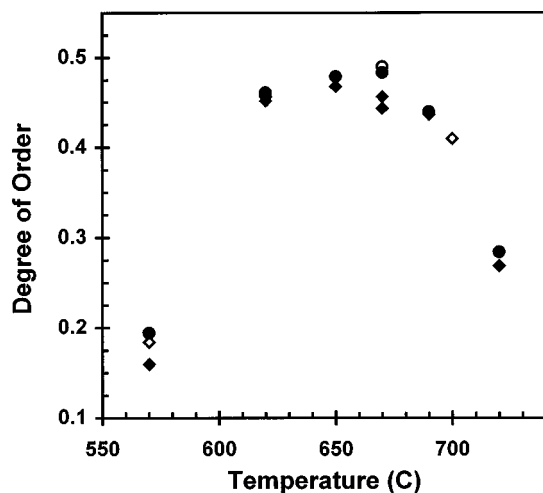


FIG. 5. Degree of order from PL peak energy vs growth temperature. Data are for: 1.5 Torr of  $\text{PH}_3$  ( $\blacklozenge$ ), 1.5 Torr of TBP ( $\diamond$ ), 3.0 Torr of  $\text{PH}_3$  ( $\bullet$ ), and 3.0 Torr of TBP ( $\circ$ ).

theless, the activation energies are consistent with experimental values of approximately 1.1 eV for (001) GaAs.<sup>16</sup> Somewhat larger values would be expected for GaInP where the bonds are stronger.

The step spacing also falls off as the temperature is increased above 670 °C. This is clearly not due to surface diffusion. However, the step spacing is known to be a function of both the surface diffusion coefficient and the sticking coefficient at the step edge.<sup>16</sup> Thus, the high-temperature behavior is attributed to a reduction in the sticking coefficient at the step edge. Again, this is qualitatively similar to the behavior observed by Asai for macroscopic islands.<sup>19</sup> He found the lateral growth rates in both the  $[110]$  and  $[\bar{1}10]$  directions to be constant at low temperatures and to decrease when the temperature was increased beyond a critical value. This was, again, schematically attributed to the loss of the singly bonded As at the  $[\bar{1}10]$  step edge (or  $[110]$  kink) at high temperatures.

The temperature dependence of the degree of order for these samples is shown in Fig. 5. The data clearly show that the degree of order has a maximum value at approximately 670 °C, with a rapid decrease as the temperature is increased or decreased. This general behavior has been reported repeatedly in the literature.<sup>10,22,27,28</sup>

The high-temperature behavior has been attributed to the loss of  $[\bar{1}10]$  P dimers with increasing temperature.<sup>27</sup> This is presumed to be due to a progressive change between the various  $(2 \times 4)$  reconstructions, with the phase formed with each increase in temperature having a lower concentration of  $[\bar{1}10]$  P dimers. Consistent with the interpretation of the effect of  $p_P$ , the change from the  $(2 \times 4)$  phase with a high concentration of  $[\bar{1}10]$  P dimers at temperatures of 670 °C and lower to the various  $(2 \times 4)$  reconstructions having lower P dimer concentrations at higher temperatures is postulated to coincide with a change in the configuration of the steps leading to lower adatom sticking coefficients at the  $[\bar{1}10]$  and  $[110]$  step edges. The alternative explanation that the decrease in the sticking coefficient at the steps leads di-

rectly to the decrease in the order parameter is, again, considered unlikely.

Again, it would appear that a reasonable interpretation of the low-temperature data is that the  $(2 \times 4)$  phase with a high concentration of  $[\bar{1}10]$  P dimers is formed at temperatures below 670 °C for a P partial pressure of 3.0 Torr, as suggested by the SPA data for growth using similar conditions.<sup>23</sup> The reduction in the surface diffusion coefficient, indicated by the reduced step spacing, acts to reduce ordering as the temperature is reduced. This kinetic explanation for the reduction in the order parameter is qualitatively consistent with the reported decrease in the order parameter with increasing growth rate.<sup>8</sup> In addition, the expected increase in the sticking coefficients at the step edges will act to reduce the degree of order.

## SUMMARY

Steps are known to affect CuPt ordering in GaInP. The way in which this occurs is still undetermined. In this study, the steps have been studied by examining the step spacing, using atomic force microscopy, as a function of the growth parameters temperature and TBP or  $\text{PH}_3$  partial pressure. The step behavior has been compared to the order parameter in the resulting epitaxial layers. A remarkable similarity in the behavior of the steps and the order parameter has been observed as the temperature and P partial pressure were changed. As the P partial pressure is reduced below a certain critical value, both the order parameter and the step spacing are observed to decrease markedly. Similarly, as the temperature is increased above a certain critical value both the step spacing and the order parameter are seen to decrease. The most likely explanation of this behavior is that at low temperatures and high values of  $p_P$  a  $(2 \times 4)$  reconstruction is formed having a high concentration of  $[\bar{1}10]$  P dimers. This surface fosters formation of the CuPt structure. Formation of this surface phase coincides with a step structure having a relatively high group III adatom sticking coefficient. As the temperature increases above the critical temperature or  $p_P$  decreases below the critical value, the surface reconstruction changes, forming  $(2 \times 4)$  phases having fewer  $[\bar{1}10]$  P dimers. This coincides with a change in the step structure resulting in a reduction in the adatom sticking coefficient. At temperatures below the critical temperature both the ordering and the step spacing are observed to decrease. This is interpreted as being mainly due to a decrease in the surface diffusion coefficient. The sticking coefficients at the step edges are expected to be increasing as well. This would lead to smaller step spacings and a lower degree of order.

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