

Step structure and ordering in Te-doped GaInP

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The step structure and CuPt ordering in GaInP layers grown by organometallic vapor phase epitaxy on singular GaAs substrates have been investigated as a function of Te (DETe) doping using atomic force microscopy, and electrical and optical properties measurements. The degree of order decreases for Te concentrations of $> 10^{18} \text{ cm}^{-3}$. It is estimated from the photoluminescence peak energy to be approximately 0.5 for undoped layers and the layers are completely disordered at sufficiently high Te doping levels. The bandgap energy is changed by 110 meV as the Te doping level increases from 10^{17} to 10^{18} cm^{-3} . The step structure also changes markedly over the range of doping that produces disordering, from a mixture of monolayer and bilayer steps for undoped layers to solely monolayer steps for electron concentrations exceeding 10^{18} cm^{-3} . For growth at 670 °C, the spacing between $[\bar{1}10]$ steps increased by over an order of magnitude as the doping level was changed over the range investigated, while the step spacing between $[110]$ steps increased only slightly. In general, Te doping significantly improves the surface morphology viewed using atomic force microscopy. The degree of order and surface structure are changed at exactly the same doping concentration. This suggests that the disordering may be controlled by the fast propagation of $[110]$ steps due to kinetic effects at the step edges. A qualitative model is presented to explain these effects. © 1998 American Institute of Physics. [S0021-8979(98)02017-9]

INTRODUCTION

Atomic-scale ordering to produce the CuPt structure frequently occurs in $\text{Ga}_{0.52}\text{In}_{0.48}\text{P}$ layers grown by organometallic vapor phase epitaxy (OMVPE) on (001)-oriented GaAs substrates.¹ The Ga and In atoms are spontaneously segregated into alternating $\{111\}\text{B}$ monolayers. Theoretically, for vapor phase epitaxy, the alternating surface stresses resulting from the formation of $[\bar{1}10]$ -oriented phosphorous dimers on the $(2 \times n)$ reconstructed (001) surface thermodynamically stabilize the variants of the CuPt structure with ordering on the $(\bar{1}11)$ and $(1\bar{1}1)$ planes.^{1,2}

This phenomenon is of considerable practical interest since ordering has a large effect on the materials properties, e.g., the bandgap energy is found to be 160 meV lower in partially ordered $\text{Ga}_{0.52}\text{In}_{0.48}\text{P}$ than in disordered material of the same composition.³ This is very important for visible light emitting diodes (LEDs) and injection laser diodes (LDs). Ordering must be avoided in order to produce the shortest wavelength devices. On the other hand, ordering offers the attractive possibility of producing heterostructures by changing the bandgap energy without altering the solid composition.³

The driving force for ordering is understood, as described above, but the mechanism remains unknown even though several speculative models have been proposed.⁴ Besides the known role of surface reconstruction, surface steps may also play an important role in the ordering process. For example, as the growth temperature is increased from 520 to 670 °C,^{5,6} the average step height (bilayer versus monolayer) and the degree of order change simultaneously. In addition,

$[\bar{1}10]$ steps are observed to assist the ordering process but $[\bar{1}10]$ steps retard ordering.⁷ These results appear to indicate that kinetic effects at step edges affect the ordering process under certain growth conditions.

One of the factors having a strong effect on ordering is doping. Several studies in GaInP have demonstrated a connection between ordering and n -^{8,9} or p -type¹⁰⁻¹⁴ dopant concentration. The results show that a significant decrease in ordering (or increase in bandgap energy) is caused by introducing a high concentration of dopants. However, the mechanism for this effect is not understood.

The purpose of this article is to present the results of a study of Te dopant effects on step structure, surface reconstruction, and ordering in GaInP. By using atomic force microscopy (AFM), the surface morphologies and step structures in Te-doped GaInP were investigated for the first time. Surface photo absorption (SPA) was used to monitor the surface reconstruction. Above a Te concentration of approximately 10^{18} cm^{-3} , a sharp drop in the degree of order was observed. This corresponds to a dramatic change in the step structure. The surface reconstruction remains unchanged. A qualitative model is proposed to explain the simultaneous change in ordering and step structure at a particular Te doping concentration.

EXPERIMENTAL PROCEDURE

Te-doped GaInP layers were grown by OMVPE in a horizontal, infrared-heated, atmospheric pressure reactor using trimethylindium (TMIn) or ethyldimethylindium (EDMIn), trimethylgallium (TMGa), and tertiarybutylphosphine (TBP) with diethyltelluride (DETe) as the dopant precursor on semi-insulating GaAs substrates with the singular (001)

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orientation. The DETe was diluted to 5 ppm in H₂. The carrier gas was Pd-diffused hydrogen. Substrate preparation consisted of degreasing followed by a 1 min etch in a 2:12:1 solution of NH₄OH, H₂O, and H₂O₂. Before beginning the GaInP growth, a 0.05 μm GaAs buffer layer was deposited to improve the quality of the GaInP layer. Two reactors were used for the growth runs. In one reactor, the temperature was 670 °C, the In precursor was TMIIn, the growth rate was approximately 0.5 μm/h, the GaInP thickness was 0.25 μm, and the TBP partial pressure and V/III ratio were kept constant at 3.0 Torr and 180, respectively. The other reactor contains a built-in optical system for surface photo absorption (SPA) measurements. In this system the temperature was 620 °C, the In precursor was EDMIn, the growth rate was 0.3 μm/h, the layer thicknesses were all 0.15 μm, and the TBP partial pressure and V/III ratio were kept constant at 0.375 Torr and 40, respectively. After completing the growth, the group III precursors were removed and the samples were cooled rapidly to room temperature.

In one reactor, the surface bonding was measured *in situ* using SPA, as described in detail in Ref. 15. The free electron concentrations and mobilities were measured at room temperature using Hall effect measurements with the Van der Pauw geometry. Ohmic contacts were formed using indium dots alloyed for 10 min. at 300 °C in N₂. The solid composition of the GaInP layers was determined using Vegard's law, from x-ray diffraction measurement using Cu Kα radiation. Only results for lattice matched layers, with values of GaP concentration in the solid of 0.515, are presented here. The 20 K photoluminescence (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. The low temperature PL peak energy was used to determine the degree of order, *S*, using the following equation:¹⁵

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

The surface morphology, including the step structure, was characterized 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 to 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, covered by a thin, conformal oxide layer.

RESULTS

Figure 1 shows the section views of the AFM images for several Te doping levels at a growth temperature of 670 °C. The addition of Te clearly produces much smoother surfaces. The AFM images show an island structure for the undoped layers. For the highly doped layers, only a few steps are observed due to the slight misorientation of the nominally singular (001) substrates. This is clearly shown in the plot of rms roughness, measured using the AFM, versus carrier concentration in Fig. 2. The effect begins at a concentration of approximately 3 × 10¹⁷ cm⁻³ and saturates for concentrations exceeding 1 × 10¹⁸ cm⁻³. The data are seen to be simi-

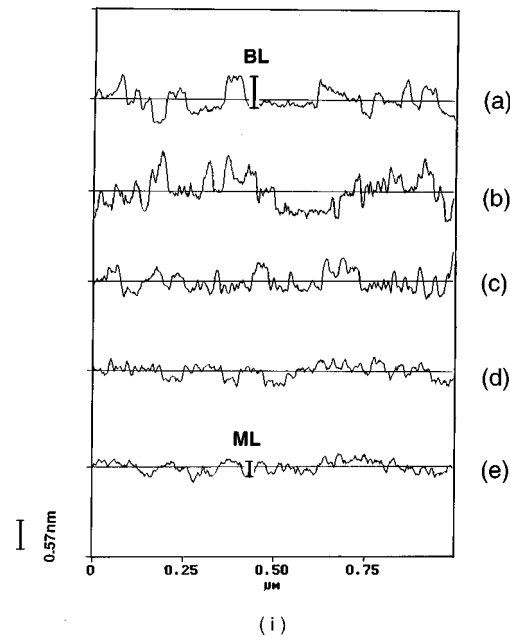


FIG. 1. (110) section atomic force microscopy scans of singular GaInP layers grown at 670 °C. DETe mole fractions are: (a) 0, (b) 2.5 × 10⁻⁹, (c) 5.3 × 10⁻⁹, (d) 1.6 × 10⁻⁸, and (e) 4.8 × 10⁻⁸. The scales indicate the heights of bilayer (BL) and monolayer (ML) steps.

lar for the two reactors at growth temperatures of 620 and 670 °C. A similar smoothing of GaInP morphology was reported for Se.⁹

Over this concentration range, increasing the Te doping causes the surface step structure to change from a mixture of monolayer and bilayer steps (33% monolayer) to completely monolayer steps, as seen in Fig. 3(a). The percentage of bilayer steps was obtained from a careful counting of twenty 1 μm AFM profiles. The layer with the highest electron density of 8.4 × 10¹⁸ cm⁻³ has defects (or microclusters), believed to be due to formation of a Te-rich second phase on the surface.

Figure 3(b) shows the degree of order, obtained from the PL data using Eq. (1), versus the Te doping level. Note that

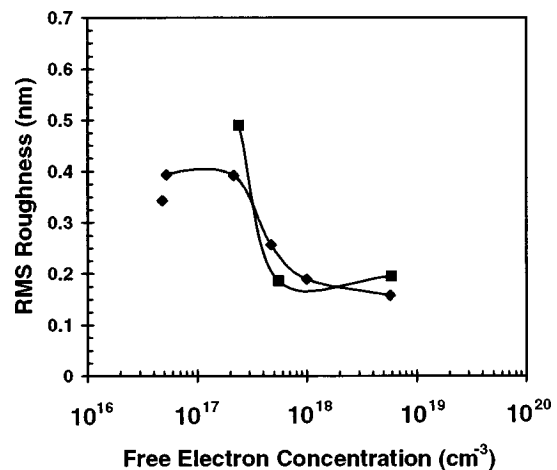


FIG. 2. rms roughness from the AFM measurements on a 1 μm × 1 μm area vs electron concentration from Te doping for growth at 620 (■) and 670 °C (◆).

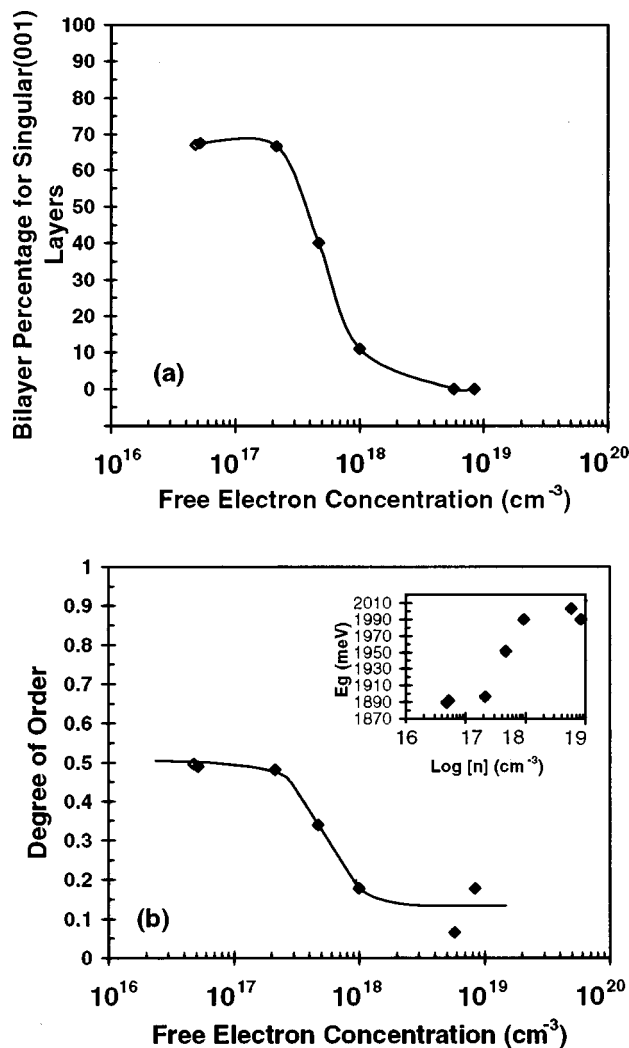


FIG. 3. (a) Percentage of bilayer steps vs free electron concentration. (b) Degree of order vs free electron concentration. Inset shows bandgap energy vs free electron concentration. All the data are for growth at 670 °C. The lines were simply drawn to fit the data points.

the samples become disordered at $\geq 1 \times 10^{18} \text{ cm}^{-3}$. These doping densities are exactly the same as those at which the step structure is observed to change. The inset in Fig. 3(b) shows the 20 K PL peak energy versus doping concentration from which the values of the degree of order were determined. The bandgap energy is abruptly increased from 1890 to around 2000 meV by the addition of Te. These carrier concentrations giving disordered layers are compared with those from other reports in the literature in Table I.

Figure 4 shows the average step spacing versus doping level for the layers grown at 670 °C. The average step spacing was obtained from a careful counting of the average step spacing along ten 1 μm AFM profiles. The spacing between [110] steps increases by approximately a factor of 20 as the doping level increases, while the step spacing between [110] steps increases only slightly. A marked elongation of islands along the [110] direction is clearly observed in the AFM images for layers grown with high Te doping concentrations. It is important to note that this effect of high Te concentrations cannot be replicated by increasing the TBP partial

pressure.¹⁶ The order parameter is virtually independent of TBP partial pressure at 670 °C for values of >3.0 Torr.

Since the surface bonding, as determined from SPA spectroscopy, has been correlated with the occurrence of CuPt ordering,^{7,15} the SPA anisotropy at 400 nm was monitored as Te was added to the system at concentrations necessary to produce disordering. The experiment was initiated by the growth of an undoped GaInP layer at 620 °C. The first 10 min of the data seen in Fig. 5 were used to determine the difference in SPA anisotropy for a group III stabilized surface and a TBP stabilized surface, using the same partial pressure used in the growth run. This difference is proportional to the concentration of $[\bar{1}10]$ P dimers on the surface.¹⁵ After 10 min, 15 sccm of DETe was added to the TBP atmosphere. This is the amount of DETe required to dope the layer to $n = 1.5 \times 10^{18} \text{ cm}^{-3}$, as indicated in a separate growth run using the same conditions. DETe was then removed from the system for 5 min, followed by the introduction of an even higher DETe concentration, with a flow rate of 25 sccm (found to produce a doping level of $n = 5.9 \times 10^{18} \text{ cm}^{-3}$). DETe was then removed from the system and the difference in anisotropy for group III and TBP stabilized surfaces was again measured to be certain that a change in the surface or the system had not compromised the results. Since this difference is similar to that at the beginning of the experiment, the results were judged to be valid.

The results, seen in Fig. 5, show that at the highest Te doping levels, no change in the SPA intensity is observed. This is taken as strong evidence that the reduction in ordering due to Te doping is not caused by a change in the surface reconstruction.

DISCUSSION

The effects of the addition of Te during the OMVPE growth of GaInP are dramatic. The $[\bar{1}10]$ step velocity, as indicated by the step spacing, increases sharply for Te concentrations exceeding 10^{18} cm^{-3} , with little change in the [110] step velocity. In addition, the step structure clearly changes from predominately bilayer to exclusively monolayer. The layers also become much smoother, with the virtual elimination of the mound or island structure. All of these changes occur with no alteration of the SPA anisotropy at 400 nm due to $[\bar{1}10]$ oriented P dimers characteristic of the $(2 \times n)$ reconstructed surface. The Te concentration at which the marked changes in step structure occur coincides with that at which the GaInP changes from highly ordered to completely disordered.

Clearly, the decrease in the degree of order is not caused by the reduction in the thermodynamic driving force for formation of the CuPt structure in Te-doped GaInP.

It is worthwhile to attempt to use the data presented to deduce something about the fundamental processes leading to a reduction in ordering at high Te concentrations. It is certain that the formation of monolayer steps is not the factor leading to a reduction in ordering. It is well documented that by changing the P precursor from TBP to PH_3 the percent of bilayer steps can be changed significantly with no change in the degree of order in the resulting layers.¹⁷

TABLE I. The carrier concentrations, above which the doped GaInP becomes completely disordered. Other factors affecting ordering are also listed. All epitaxial layers were grown by OMVPE.

Carrier concentration (cm ⁻³)	Dopant	Substrate misorientation	Temperature (°C)	V/III ratio	Method of measurement	References
10 ¹⁸	Te	(001)	670	180	PL	This Report
6 × 10 ¹⁷	Te	3° off (111)B	670	180	PL	29
3.8 × 10 ¹⁷	Si	2° off (111)A	700	457	PL, TEM	8
2 × 10 ¹⁸	Se	2° off (111)A	700	118–132	PL, TEM	8
10 ¹⁹	Se	2° off (111)A	670	100	TEM	9
3 × 10 ¹⁸	Se	2° off (111)A	740	100	TEM	9
10 ¹⁸	Zn	2° off (111)A	675	65	Absorption coefficient	14
2 × 10 ¹⁸	Zn	(001) or 2° off (111)A	660–700	130–230	PL and/or TEM	10, 12, 13
2 × 10 ¹⁸	Mg	2° off (111)A	680	140	PL	10

One factor known to destroy CuPt ordering in GaInP layers is the increase in the group III self-diffusion coefficients due to the presence of high concentrations of the dopant Zn. Annealing for prolonged times at high temperatures leads to the elimination of ordering in layers grown having the CuPt structure.¹⁸ Several factors indicate that this is not the mechanism for the disordering induced by Te in this study. (1) The times and temperatures involved in this study are unlikely to result in the complete disordering of the layers. (2) The disordering process is virtually the same for growth at 620 and 670 °C. (3) It would be a remarkable coincidence that the step structure changes so dramatically at exactly the Te doping concentration leading to disordering due to diffusion in the “bulk” layers. All of these factors suggest that it is, in fact, the change in step structure that leads to the suppression of ordering at the surface during growth.

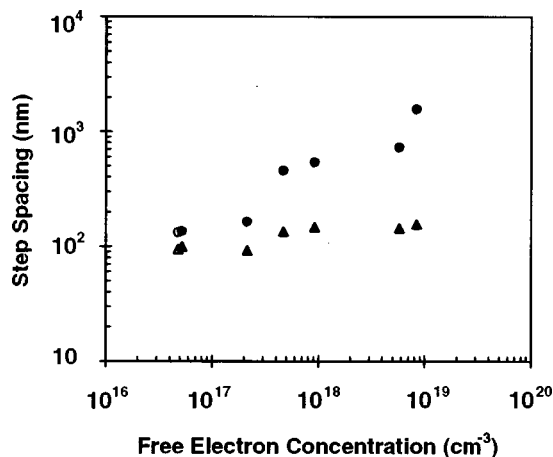


FIG. 4. Step spacing vs electron concentration for GaInP layers grown at 670 °C with intentional Te doping (filled symbols) and undoped (open symbols): (●,○) for [110] steps and (▲,△) for [111] steps.

The most likely explanation for the effects reported here is the effect of Te on the step structure and, consequently, the attachment of group III adatoms at the step edge during growth. Clearly, since formation of the CuPt structure is driven by the surface thermodynamics, it requires the ability of Ga and In atoms to exchange positions at the step edge. This is facilitated by small sticking coefficients. In the extreme case, group III adatom sticking coefficients of unity would result in a completely random alloy. The effect is very

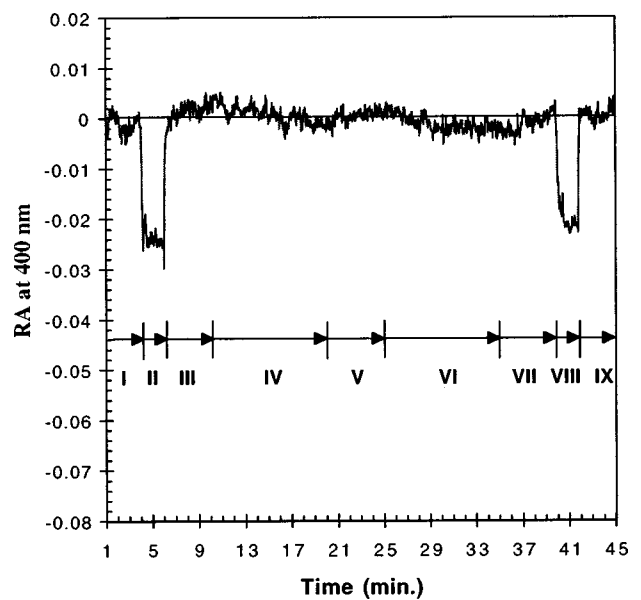


FIG. 5. SPA anisotropy ($R[\bar{1}10]-R[110]$) measured at 400 nm, RA(400 nm), showing the effect of adding DETe on the (001) surface structure of GaInP layers grown at 620 °C. The switching sequences of the sources are: I-TBP in for 4 min, II-TBP out for 2 min, III-TBP in for 4 min, IV-15 sccm DETe ($n=1.5 \times 10^{18} \text{ cm}^{-3}$) for 10 min, V-DETe out for 5 min, VI-25 sccm DETe ($n=5.9 \times 10^{18} \text{ cm}^{-3}$) in for 10 min, VII-DETe out for 5 min, VIII-TBP out for 2 min, IX-TBP in for 4 min.

strong for a relatively small Te concentration, suggesting that Te collects at the $[\bar{1}10]$ step edges, acting as a special sort of surfactant.

Simple electron counting arguments may be useful as a guide to the behavior of Te on the surface and at step edges. These simple criteria have been used to rationalize the formation of the (2×4) reconstructed structure on (001) surfaces.¹⁹ In the bulk solid, each P atom contributes $5/4$ of an electron to each of the four sp^3 bonds formed. On the (001) GaInP surface, the P makes two back bonds to group III atoms, involving $2 \frac{1}{2}$ electrons. The $[\bar{1}10]$ P dimer bonds involve another electron and a lone pair is formed on each surface P atom. This totals more than the 5 valence electrons on each P atom. However, every fourth P dimer is missing in the (2×4) structure,¹⁹ which accounts for all of the electrons on the surface atoms. Replacement of P by a Te atom disrupts this scheme. In the simplest picture, the Te atoms simply do not participate in dimerization, but form two lone pairs per Te atom.

At a $[\bar{1}10]$ step edge, the model of Asai,²⁰ which does not consider reconstruction, suggests that at high group V partial pressures, a row of singly bonded group V atoms forms. In reality, this is extremely unlikely. The actual atomic structure is not known; however, assuming bonding processes similar to those on the (001) surface, it seems likely that dimer bonds will form between the P atoms at the step edge to reduce the energy. Electron counting constraints can be satisfied by several structures with the formation of few, if any, dangling bonds. This would account for the relatively low group III adatom sticking coefficients at the $[\bar{1}10]$ step edge, even for the high partial pressures of the P precursor where the step is apparently saturated with the "dangling" P atoms.¹⁶ The factor of 20 increase in step spacing induced by the addition of Te supports this assertion. The presence of Te at the $[\bar{1}10]$ step edge clearly acts to markedly increase the adatom sticking coefficient, as evidenced by the $20 \times$ increase in the $[\bar{1}10]$ step spacing. This probably indicates that Te substitutes for P at the step edge with the formation of a dangling bond, due to the extra valence electron. Of course, this increase in sticking coefficient would also act to decrease the degree of order in the resulting layer, as discussed above. This explanation is speculative, of necessity, since the actual step structure cannot be reliably measured in an OMVPE reactor.

The increased group III adatom sticking coefficient at the step edge accounts for the increased step spacing and the consequent smoothing of the surface morphology. The change from predominantly bilayer to monolayer steps with increasing Te concentration could be due to either thermodynamic or kinetic factors. A change in the step structure by the collection of Te atoms may result in a reduction in the driving force for formation of the (2×2) structure at the $[\bar{1}10]$ step edge.²¹ The formation of bilayer $[\bar{1}10]$ steps was previously attributed to the formation of a (2×2) reconstruction at the bilayer step edge.²² On the other hand, a change in the ratio of "up" and "down" sticking coefficients is known to affect the step morphology. When this ratio is high, the formation of an ordered array of monolayer steps is expected.²³ This would imply that the sticking coef-

ficient at up steps is increased much more than for the down steps by the addition of Te at the step edges.

Another possible effect of high concentrations of Te donors on ordering should also be considered. Rather than changing the density of $[\bar{1}10]$ P dimers, the donor could result in the dimers not being aligned into $[110]$ rows. Just such an effect has been observed for the donor Si on the surface structure of GaAs. Pashley²⁴ found a high concentration of kinks, i.e., a mismatch between the phase of P dimers in adjacent (2×4) unit cells, at high doping concentrations. This would not remove the driving force for ordering, but would produce an enormous number of antiphase boundaries (APBs). The layers grown with high Te doping levels have been examined using transmission electron microscopy. The addition of Te has been found to have essentially no effect on the density of APBs.²⁵ In addition, the presence of APBs would not be expected to increase the PL peak energy, as observed in the highly Te doped samples. Thus, this mechanism is tentatively rejected as an explanation of the observations reported here.

The addition of the dopants C, O, Si, and Mg, with smaller atomic weights, during the OMVPE growth of GaAs was found to roughen the surface for doping concentrations exceeding 10^{18} cm^{-3} .^{26,27} The roughening was interpreted as due to the preferential attachment of dopants at the step edges blocking the attachment of host adatoms. The resulting reduction of the step mobility was postulated to yield the rough surfaces observed.^{26,28} The addition of Zn and Se, dopants with larger atomic weights, caused the GaAs surface rms roughness to change very little for doping concentrations as high as 10^{19} cm^{-3} .²⁷ The weaker impurity-host atom bond strength was proposed as the key factor that prevented these impurities from blocking step motion. In the work presented here, the surface morphology of Te-doped GaInP was markedly improved by the addition of Te, in agreement with earlier Se doping studies.⁹ This phenomenon was not observed in the GaAs studies.

SUMMARY

Step structure and CuPt ordering have been investigated in GaInP grown on singular substrates as a function of Te doping. The degree of order decreases dramatically with increasing Te concentration, with a value of approximately 0.5 for undoped epitaxial layers and 0 (totally disordered) for layers with doping levels of $\geq 1 \times 10^{18} \text{ cm}^{-3}$. This corresponds to a measured increase in bandgap energy of 110 meV as the Te doping level was increased from 10^{17} to 10^{18} cm^{-3} . The surface structure is also changed dramatically by the addition of Te over the range that produces disorder: (i) The islands or mounds observed on the surface of undoped layers disappear when the Te doping exceeds 10^{18} cm^{-3} . This results in a marked decrease in the rms roughness. (ii) The bilayer steps seen for undoped layers disappear for electron concentrations of $\geq 1 \times 10^{18} \text{ cm}^{-3}$. All of the steps are a single monolayer in height. (iii) The $[\bar{1}10]$ step spacing increases by a factor of 20 with increasing Te concentration.

The degree of order and surface structure were observed to change at exactly the same doping concentration. This suggests that the ordering phenomenon may be retarded by the rapid propagation of $[110]$ steps due to kinetic effects at the step edges. Increasing the concentration of the dopant, DETe, was found to have no effect on the SPA signal at 400 nm, a measure of the concentration of $[110]$ P dimers. This suggests that the effect of Te on ordering is not simply due to a reduction in the surface thermodynamic driving force for ordering. A qualitative model based on the electron counting rule has been proposed to explain the effect of Te on adatom attachment at step edges.

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