

Surfactant controlled growth of GaInP by organometallic vapor phase epitaxy

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The effect of the surfactant Sb has been studied for GaInP semiconductor alloys grown by organometallic vapor phase epitaxy. Dramatic changes in the optical and electrical properties of GaInP with CuPt ordering have been observed. A small concentration of triethylantimony (TESb) in the vapor is found to cause Sb to accumulate at the surface. *In situ* surface photoabsorption analysis indicates that Sb changes the surface bonding by replacing the $[\bar{1}10]$ P dimers that are responsible for the formation of the CuPt structure during growth with $[\bar{1}10]$ Sb dimers. As a result, the degree of order for the GaInP layers is decreased, as shown by transmission electron diffraction studies. The 20 K photoluminescence spectra show a 131 meV peak energy increase for GaInP layers grown on vicinal substrates when a small amount of Sb $[\text{Sb}/\text{P}(v) = 4 \times 10^{-4}]$ is added to the system during growth. The use of surfactants to control specific properties of materials is expected to be a powerful tool for producing complex structures. In this article, the growth of heterostructures by modulating the Sb concentration in the vapor is demonstrated. © 2000 American Institute of Physics. [S0021-8979(00)05008-8]

I. INTRODUCTION

The use of surfactants during vapor phase epitaxy (VPE) processes is beginning to be studied for both the group IV elemental¹ and III/V compound semiconductors.^{2,3} The word surfactant is defined as a surface-active substance.⁴ In VPE growth, a surfactant typically refers to a substance which accumulates at the surface and alters the surface properties of the material. Generally, a material with a low solubility and a low vapor pressure is most likely to build up on the surface, since it is rejected from the lattice, but is not evaporated. This will produce a high surface concentration that can modify the growing surface in several ways. For example, the surfactant may modify the bonding at the surface resulting in changes in the surface energy¹ and the growth process at or near the surface.²

To date, most reported surfactant effects for semiconductors relate to the morphology of highly strained layers grown by molecular-beam epitaxy (MBE). The addition of dopants during MBE growth of group IV semiconductors has been shown to affect both adatom attachment at step edges^{5,6} and surface reconstruction.^{7,8} Dopants, such as Sb, As, and Te, have been shown to modify the growth mode of Ge on Si (001) surfaces.² Sb has also been shown to destroy the ordering in SiGe alloys by changing the surface reconstruction during MBE growth.⁸ Recently, the surfactant Bi has been used to improve the crystalline quality of $\text{Si}_{1-x}\text{Sn}_x$ layers grown by MBE.⁹

In III/V semiconductors, the isoelectronic elements As, Sb, and Bi also act as surfactants.¹⁰⁻¹² Arsenic has been

shown to modify the surface reconstruction of cubic GaN grown by MBE.¹⁰ In addition, Bi has been shown to modify the surface morphology of wurtzite GaN films¹¹ and Sb is found to improve the optical properties of AlGaAs grown by MBE.¹²

A similar surfactant effect is expected for organometallic vapor phase epitaxy (OMVPE). For example, Sb has been found to accumulate at the interface during the OMVPE growth of InAs/InPSb and InAs/AlSb multiquantum wells.¹³

The effects of surfactants can be much more dramatic in semiconductor alloys with CuPt ordering, including large changes in the electrical and optical properties.¹⁴ The change in CuPt order parameter induced by the surfactant translates into a marked change in the band-gap energy.

The CuPt ordered structure with ordering on $\{111\}$ planes is typically formed in $\text{Ga}_{0.52}\text{In}_{0.48}\text{P}$ layers grown on singular (001) GaAs substrates by OMVPE.¹⁵ CuPt ordering is of practical interest, since it has a significant effect on the materials properties, e.g., the band-gap 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.¹⁶ For visible light emitting diodes and injection laser diodes, it is important to avoid ordering in order to produce the shortest wavelength devices. The ordering phenomenon is also of fundamental interest in terms of the thermodynamics of III/V alloys and surface processes occurring during VPE. The thermodynamic driving force for CuPt ordering in GaInP originates from formation of $[110]$ rows of $[\bar{1}10]$ -oriented P dimers on the (2×4) reconstructed (001) surface.¹⁷ The resulting surface stresses stabilize the two variants of the CuPt structure with ordering on the $(\bar{1}11)$ and $(1\bar{1}1)$ planes.¹⁷ The degree

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of order has been found to be reduced by decreasing the $[\bar{1}10]$ P dimer concentration on the surface. This was demonstrated by increasing the temperature and by decreasing the partial pressure of the group V precursor during the OMVPE growth of GaInP.^{18,19} In addition, kinetic effects at the surface steps may be important in the ordering process. For example, $[110]$ steps have been observed to assist the ordering process, but $[\bar{1}10]$ steps retard ordering.²⁰

One of the factors having a strong effect on ordering is doping. Several studies with various dopants, *n*-type^{21–25} and *p*-type,^{26–30} have found a connection between ordering and doping concentration in GaInP. For Si²¹ and Zn,³⁰ the effect was attributed to enhanced Ga/In diffusion in the bulk. For Te, the disordering mechanism is different. The addition of Te was observed to increase the $[\bar{1}10]$ step velocity by a factor of 20 \times , attributed to a marked change in group III adatom attachment kinetics at the step edge.^{23–25} The coordinated change in CuPt order parameter and step structure suggests that the change in step structure causes the change in ordering. This is one of the few examples of surfactant effects during OMVPE growth. The effect of the surfactant Te on the band-gap energy raises the possibility of producing heterostructures and elaborate multilayer structures, required for the most advanced devices, by simply modulating the concentration of a surfactant during growth.³¹ This would be particularly powerful if the surfactant did not result in the loss of control of the Fermi level position in the structure.

The primary objective of the work reported here was to study the effects of the isoelectronic dopant Sb on ordering in GaInP grown by OMVPE. Sb is expected to act as a surfactant: It has a small solubility in GaInP, since Sb is much larger than P.^{32,33} In addition, Sb is less volatile than P. Therefore, Sb is likely to accumulate at the surface during growth. Another objective was to demonstrate a useful new technique for controlling the band gap and other properties of semiconductors by modifying the surface structure during growth.

II. EXPERIMENT

The Ga_{0.52}In_{0.48}P epilayers in this work were grown lattice matched to both singular and vicinal (misoriented by 3° toward the $(111)B$ direction) GaAs substrates by OMVPE in a horizontal, atmospheric pressure apparatus using tertiary-butylphosphine (TBP), trimethylgallium (TMGa), and ethyldimethylindium (EDMIn) precursors. Triethylantimony (TESb) was used as the Sb precursor. The carrier gas was Pd-diffused hydrogen. The growth temperature and the V/III ratio were kept at 620 °C and 40, respectively. The GaInP layer thickness was approximately 0.4 μm with a growth rate of 1.2 $\mu\text{m}/\text{h}$. The Sb molar flow rate was varied from 0 to 1.32×10^{-7} mole/min.

A surface photoabsorption (SPA) system attached to the OMVPE reactor was used for *in situ* measurements. P-polarized light from a 150 W Xe lamp was used to irradiate the GaInP surface at an incidence angle of approximately 70° through a polarizer and a chopper. The reflected light was dispersed with a compact monochromator and detected

with a Si PNN⁺ photodiode using standard lock-in amplifier techniques.

The SPA measurement procedure was as follows. A 0.4 μm ordered Ga_{0.52}In_{0.48}P layer without Sb was grown first on a singular (001) GaAs substrate at 620 °C. Then, the reflectivity of the group V terminated surface was taken with TBP flowing through the reactor. The wavelength scan was from 350 to 800 nm. After the scan, TBP was switched out of the chamber and the surface was allowed to stabilize for 2 min. The reflectivity of the group III terminated surface was then measured over the same wavelength range. To recover the P terminated surface, TBP was first switched back into the reactor for 6 min and the reflectivity was measured again. The reflectivity of the recovered surface was compared with that of the initial P stabilized surface to ensure that it did not degrade during measurement of the group III terminated surface at 620 °C. Previously published SPA measurements¹⁴ were made after cooling to 520 °C, because of a concern that the surface would degrade. However, SPA measurements at 620 °C are preferred, because they provide information about the surface at the growth conditions.

TESb with a fixed flow rate $[\text{Sb}/\text{P}(v) = 4 \times 10^{-4}]$ was added to the reactor with TBP for 15 min without growth. The reflectivity of the P plus Sb terminated surface was measured again. This procedure was performed with the incident light parallel to the $[110]$ and then the $[\bar{1}10]$ direction. The difference between the SPA reflectivity of group V and group III terminated surfaces, normalized by the group III reflectivity, is termed the SPA difference spectrum. The SPA anisotropy spectrum is the difference between the $[\bar{1}10]$ and $[110]$ SPA difference spectra. The SPA anisotropy is $\{[R(V) - R(\text{III})]/R(\text{III})\}_{[\bar{1}10]} - \{[R(V) - R(\text{III})]/R(\text{III})\}_{[110]}$, where $R(V)$ and $R(\text{III})$ are the reflectivity of the group V and the group III stabilized surface, respectively.

The 20 K photoluminescence (PL) was excited by the 488 nm line of an Ar⁺ laser with a power of 10 mW focused to a 0.5 mm² spot. The signal was dispersed with a SPEX monochromator and detected with a Hamamatsu R1104 head-on photomultiplier. The degree of order (S) was calculated from¹⁸

Degree of Order (S)

$$= \sqrt{\frac{2005 - \text{PL peak energy at 20 K (in meV)}}{471}},$$

where 2005 meV is the band-gap energy of completely disordered GaInP and 471 meV is the maximum change in the band-gap energy for perfectly ordered GaInP.

$[110]$ 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 a JEM 2010 scanning TEM operated at 200 kV.

III. RESULTS

The 20 K PL spectrum for an ordered Ga_{0.52}In_{0.48}P single layer grown on a vicinal substrate without Sb occurs at 1840.8 meV. The PL spectrum for a Ga_{0.52}In_{0.48}P single

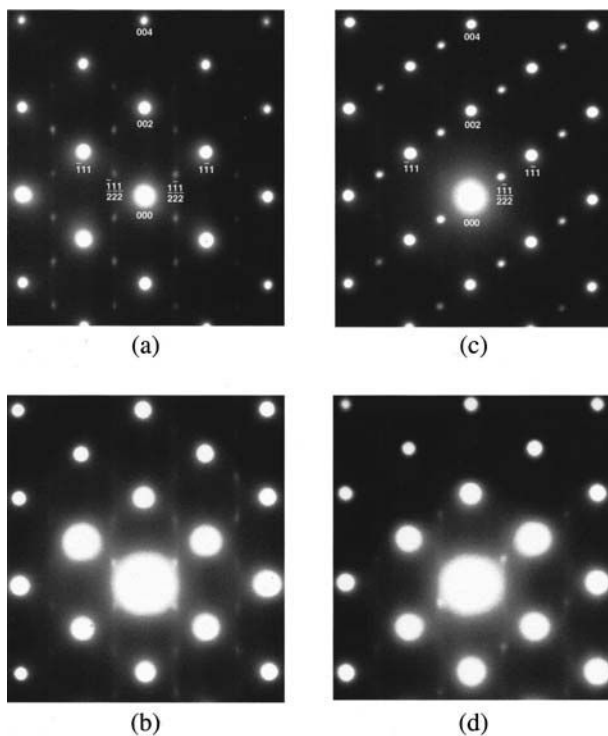


FIG. 1. TED patterns for undoped, ordered GaInP layers on vicinal (3°_B) (c) and singular (a) GaAs substrates and for disordered layers grown with the addition of Sb [$Sb/P(v) = 4 \times 10^{-4}$] on (d) vicinal and (b) singular substrates.

layer on a vicinal substrate with the addition of Sb (Sb/P ratio in the vapor phase of 4×10^{-4}) shifts by 131 MeV to 1971.8 meV. The PL indicates the layers are highly ordered without Sb ($S=0.59$) and much more disordered when grown with Sb present ($S=0.27$).

The PL results are confirmed by the TED data. Figures 1(d) and 1(c) show the $[110]$ pole TED patterns for GaInP on vicinal substrates with and without Sb, respectively. As seen in Fig. 1(c), sharp spots are obtained at the $1/2(1\bar{1}1)$ position in addition to the normal zinc-blende lattice spots. The spots are very intense, consistent with the high degree of order ($S=0.59$). However, the CuPt superspots essentially disappear in Fig. 1(d). This indicates that the layer is almost completely disordered. Clearly, the relative intensities of the order superspots are dramatically decreased when Sb is added. The results are similar for GaInP layers grown on singular substrates using the same growth parameters. The PL peak shifts about 95 meV and the relative intensities of the ordered spots decrease when Sb is present as seen in Figs. 1(a) and 1(b). Note that the difference between the ordered and disordered layers is less for layers grown on singular substrates than for those grown on vicinal substrates. In addition, only one variant is observed on vicinal substrates due to the presence of $[110]$ steps caused by the substrate misorientation.³⁴

The PL peak energy is plotted versus the Sb/P ratio in the vapor phase in Fig. 2. The peak energy is observed to increase with an increase in the input amount of Sb. The data show that a small amount of Sb in the vapor phase ($Sb/P = 4 \times 10^{-4}$) is sufficient to markedly decrease the ordering.

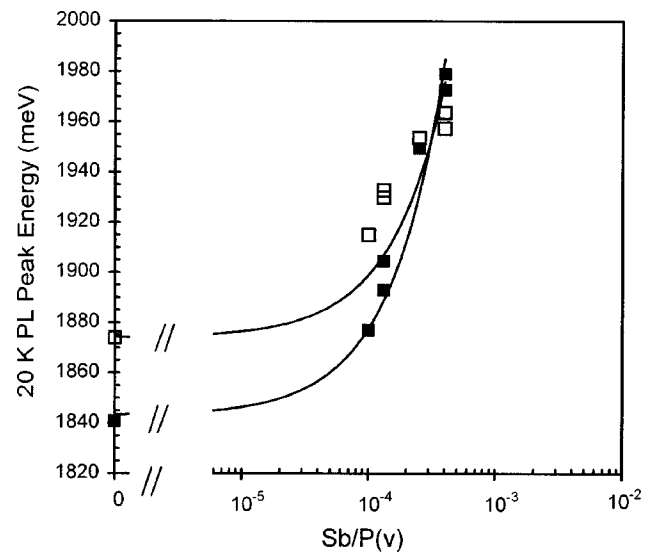


FIG. 2. 20 K PL peak energy vs the Sb/P ratio in the vapor phase for GaInP grown on (□) singular and (■) vicinal substrates.

Figure 3 shows the SPA anisotropy spectra for GaInP with and without Sb on singular GaAs substrates. For ordered GaInP, the positive peak at about 400 nm is attributed to P dimers aligned along the $[\bar{1}10]$ direction.³⁵ The magnitude of this quantity is proportional to the $[\bar{1}10]$ P dimer concentration, $[P_{[\bar{1}10]}^2]$.³⁵ This surface is referred to as “ (2×4) like,” since the long range periodicity cannot be determined from optical measurements. After Sb was added for 15 min with an Sb/P ratio of 4×10^{-4} , the intensity of the SPA anisotropy signal at 400 nm dropped to nearly zero. This indicates that Sb eliminates the P dimers. It is interesting to note that a broad peak at around 700 nm also appears when Sb is added. This may be related to $[\bar{1}10]$ Sb dimers, as discussed below.

Figure 4 shows the degree of order and SPA anisotropy at 400 nm versus the Sb/P ratio. In this figure, the degree of order (S) for various Sb flow rates was deduced from the 20 K PL peak energy. The results indicate that the degree of

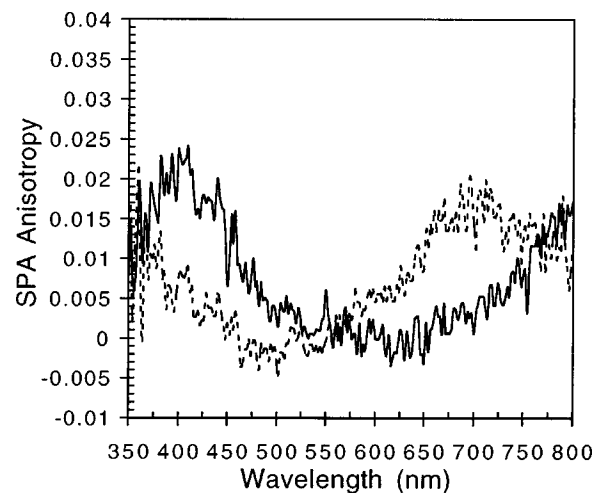


FIG. 3. SPA anisotropy spectra for GaInP layers grown with (dashed line) and without Sb (solid line).

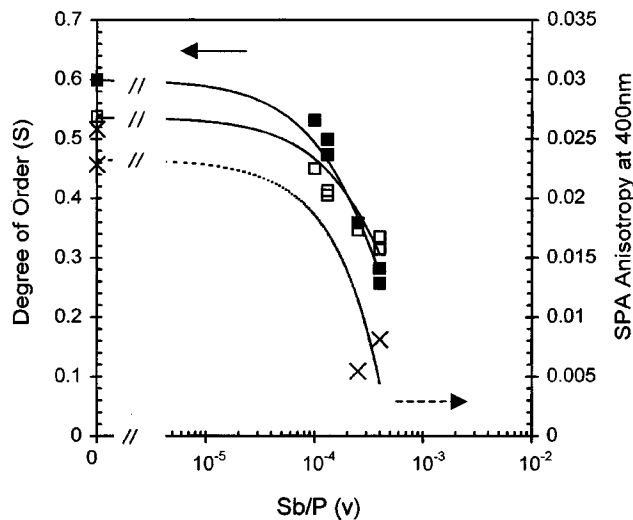


FIG. 4. SPA anisotropy signal at 400 nm (x) for singular substrates and the degree of order (S) vs Sb/P ratio in the vapor phase for GaInP grown on singular (\square) and vicinal (\blacksquare) GaAs substrates.

order is strongly correlated to the SPA anisotropy at about 400 nm. It appears that Sb affects the ordering in GaInP by eliminating the P dimers during growth.

The ability to control the band-gap energy of GaInP by simply modulating the Sb flow rate suggested the possibility of producing heterostructures and quantum wells. A disorder-on-order (D/O) GaInP heterostructure with 0.4 μm thick layers was grown on a vicinal GaAs substrate. In the dark-field TEM image, shown in Fig. 5 the more ordered layer appears brighter. To produce a sharp interface, the ordered layer (grown first) was exposed to TBP and TESb for 5 min, during a growth interruption, to allow Sb to accumulate on the surface. In this way, when the second GaInP layer was grown, it was immediately disordered. It can be seen that the D/O interface is well defined and abrupt.

The PL spectrum from the D/O heterostructure consists of two peaks from the more and less ordered layers. A significant PL peak separation of 135.1 meV between the two layers is seen. The 135 meV band-gap discontinuity is more

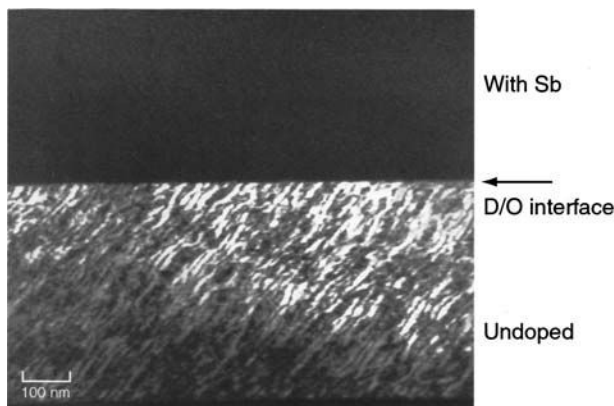


FIG. 5. [110] pole TEM cross section of a disorder-on-order GaInP heterostructure grown on a vicinal substrate where the lower layer was undoped and the upper layer was grown with a Sb/P ratio in the vapor of 4×10^{-4} . A well-defined order-on-disorder interface is observed, as indicated.

than 5 kT at room temperature, which should be sufficient for many devices. The PL spectra together with the TEM results definitively demonstrate the potential of this technique to modulate the materials properties for III/V semiconductor alloys.

IV. DISCUSSION

The effect of the surfactant Sb on GaInP layers grown by OMVPE has been studied. The results for GaInP with CuPt ordering are dramatic. As seen in Fig. 2, the addition of a tiny amount of Sb produces a marked increase in the low temperature PL peak energy. From previous studies, the Sb concentration in the solid is expected to be approximately 10^{-4} for a Sb/P ratio of 4×10^{-4} in the vapor phase.³³ This small change in solid composition of disordered GaInP due to Sb would result in a decrease in the band-gap energy of less than 1 meV.³³ Therefore, the large change in the PL peak energy presented here is not due to the small change in solid composition.

As demonstrated by TED, the PL peak shift is related to the change in ordering when Sb is added. From the SPA results, it appears that Sb removes the thermodynamic driving force for CuPt ordering in GaInP during growth, i.e., the $[\bar{1}10]$ P dimers are eliminated by the addition of Sb at concentrations sufficient to produce the growth of completely disordered layers.

The disordering mechanism seems unlikely to be a bulk effect related to an increase in the Ga and In diffusion coefficients in the solid. Since Sb is isoelectronic with P, it produces no significant change in the position of the Fermi level at the growth temperature. Thus, Sb should have essentially no effect on the native lattice defects that assist Ga/In inter-diffusion.

The disordering mechanism due to Sb addition during GaInP growth is different than that observed for Te, another surfactant. Addition of Te during GaInP growth produces a marked change in the step structure. The $[\bar{1}10]$ step velocity was increased dramatically leading directly to the loss of CuPt ordering.^{23,24} The donor Te apparently affects the adatom attachment kinetics at steps on the (001) surface.²⁵ A preliminary examination of the GaInP:Sb surface by atomic force microscopy (AFM) shows that Sb does not change the surface morphology significantly.³⁶ Of course, the situation is expected to be different for Sb since it is isoelectronic, while Te is a donor in GaInP.

As shown in Fig 3, there is a positive, broad peak near 700 nm in the SPA anisotropy spectra when Sb is added to the system during growth. This is suspected to be due to Sb dimers aligned along the $[\bar{1}10]$ direction. However, there is no published SPA information for any Sb-related compound. Kobayashi *et al.*³⁷ suggested that the optical transition energy of surface dimers measured by SPA could be estimated using Harrison's model if the dimer length were known. Generally, good agreement between the covalent energies of dimer bonds calculated from Harrison's model and the SPA anisotropy peak energies is obtained for arsenides and phosphides. It is expected that, by knowing the Sb dimer length,

the SPA anisotropy peak energy related to the Sb dimer can be predicted in this way.

It is reasonable to assume that the Sb dimer length on a GaAs surface is similar to that on $\text{Ga}_{0.52}\text{In}_{0.48}\text{P}$, since GaAs and $\text{Ga}_{0.52}\text{In}_{0.48}\text{P}$ have the same lattice constant. Sugiyama *et al.*³⁸ determined the Sb dimer bond length to be about 2.95 Å on an Sb terminated (001) GaAs surface having the (2 × 4) reconstruction. Srivastava and Jenkins³⁹ and Schmidt and Bechstedt⁴⁰ also calculated the Sb dimer bond length on (001) GaAs and obtained values of 2.79 and 2.86–2.87 Å, respectively. According to Harrison's model, the optical transition is expected to occur at approximately 650 nm for Sb dimers with a 2.95 Å bond length. Our SPA results show a broad positive peak at 700 nm. This suggests that the peak is related to the Sb dimers, although this would mean that the Sb dimer on GaInP is longer than on a GaAs surface. The SPA anisotropy spectrum taken at 520 °C, reported previously for GaInP with Sb, did not show a peak at 700 nm.¹⁴ The lower temperature likely resulted in an increase in the Sb concentration on the surface. At high Sb concentration, it has been shown that the surface reconstruction begins to change, reducing the number of Sb dimers oriented along $[\bar{1}10]$ direction.⁴¹

A disordered GaInP epilayer with a narrow PL peak is obtained by simply adding a small amount of Sb during OMVPE growth. This is an attractive way to produce disordered GaInP. Previously this has been accomplished by growing at higher temperatures¹⁸ or using highly misoriented substrates.²⁰

V. CONCLUSIONS

The effects of the isoelectronic surfactant Sb have been studied for GaInP with CuPt ordering grown by OMVPE. The 20 K PL peak energy for GaInP is increased as a small concentration of Sb is added to the vapor. The addition of Sb nearly destroys the CuPt ordering, as judged from the TED patterns.

The disordering mechanism is not a bulk effect, since adding Sb does not change the Fermi level position, and thus produces no increase in the Ga/In bulk diffusion. Neither is the step structure changed significantly by Sb. However, the SPA analysis demonstrates that the addition of Sb destroys the $[\bar{1}10]$ P dimers responsible for CuPt ordering.

The surfactant Sb has been used to control the band-gap energy of an epitaxial III/V semiconductor. The ability to independently modulate the band gap and the Fermi level is the key advantage of using the isoelectronic surfactant Sb. Heterostructures with a change in band-gap energy and no change in solid composition can be produced by simply modulating the Sb flow. A well-defined disorder-on-order heterostructure with a 135 meV band-gap difference between the two layers was produced. This technique is expected to find application as a new and powerful method for producing complex structures, such as heterostructures and quantum wells, during epitaxial growth of III/V alloy semiconductors.

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