SURFACE PHASE SEPARATION AND ORDERING IN COMPOUND SEMICONDUCTOR ALLOYS

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ABSTRACT

The orientation dependence of phase separation has been examined in detail in InGaAsP layers grown by liquid phase epitaxy on (001), (110), $(111)_{In}$ and (123) InP substrates. It is shown that phase separation is two-dimensional in nature and does not occur along the growth direction for the cases examined. Further, phase separation takes place along the soft directions lying in the growth plane. These results very strongly suggest that phase separation evolves at the surface while the layer is growing.

CuPt-type ordering characteristics of InGaAsP layers are presented. In addition, the influence of growth temperature and growth rate on domain sizes have been investigated in $GaInP_2$ layers. A model has been proposed to rationalize the formation of domains and involves steps present on the surface. Results suggest that ordering like phase seperation occurs at the surface while the layers is being deposited. It is inferred that the two microstructural features evolve concomitantly at the surface during layer growth.

INTRODUCTION

It is now well accepted that atoms in epitaxial layers of ternary and quaternary III-V compound semiconductors are not distributed at random within their respective sublattices. These deviations from randomness are manifested in two ways: (i) phase separation, and (ii) long range atomic ordering. Based on bulk thermodynamic calculations, de Cremoux et al. [1] were the first to suggest that InGaAsP materials phase separate. Subsequent calculations by Stringfellow [2] and Onabe [3] are consistent with the work of de Cremoux et al. [1].

Henoc et al. [4] were the first to observe by transmission electron microscopy (TEM) phase separation in InGaAsP epitaxial layers grown by liquid phase epitaxy (LPE) on (001) InP substrates. They have seen two types of contrast modulations: (i) coarse modulations with a periodicity of ~ 150nm, oriented along the <100> directions lying in the (001) growth plane, and (ii) fine scale speckle structure, also oriented along the <100> directions with a period of ~ 15nm. In the presence of the two types of overlapping microstructural features it is very difficult to discern compositional differences associated with the coarse contrast modulations. Since the wavelength of the coarse modulations is too large for it to evolve by bulk diffusion, Launois et al. [5] have suggested that they develop by surface diffusion, tacitly ignoring the presence of the fine scale structure.

Following the work of Henoc et al. [4], Mahajan et al. [6] have examined the microstructures of

InGaAsP layers of different compositions grown on (001) InP substrates by LPE. They have found that the wavelength of the fine scale structure is independent of the layer composition. In addition, they see rectilinear boundaries in layers grown by near equilibrium LPE. Coarse contrast modulations, resembling a basket-weave pattern are also observed. They have suggested that the fine scale structure evolves by phase separation, whereas coarse modulations could form to accommodate strain effects associated with the speckle structure. The wavelength of the coarse modulations seems too large to evolve even by surface diffusion at the growth temperature.

The subsequent studies by Chu et al. [7] and Norman and Booker [8] on microstructures of InGaAsP layers grown by vapor phase epitaxy (VPE) and LPE, respectively, have shown that when the (001) layers are examined in cross-section using the 004 reflection, i.e., the reflection parallel to the growth direction, contrast characteristic of phase-separated microstructures is not observed. This implies that there is no phase separation along the growth direction, strongly suggesting that phase separation is two-dimensional in nature. In addition, Chu et al. [7] have suggested that the fine scale speckle structure could evolve by surface phase separation at the growth temperature. On the other hand, Norman and Booker [8] have interpreted their results differently. They attribute the absence of contrast for the 004 reflection to strain effects and have argued that coarse contrast modulations result from surface phase separation. They have proposed that the fine scale structure is a consequence of phase separation occurring in the bulk during cool down from the growth temperature.

It is apparent from above that two schools of thought have emerged on the issue of phase separation. One believes that the coarse and fine contrast modulations are, results of surface and bulk phase separation, respectively. The second group [6,7] has argued that the fine scale speckle contrast develops by phase separation occurring at the surface during growth [7].

Ordering in III-V compounds was first seen by Kuan et al. [9] who observed CuAu-I type order in GaAlAs epitaxial layers grown on (110) GaAs substrates by molecular beam epitaxy (MBE) and organo-metallic vapor phase epitaxy (OMVPE). Similarly ordered structures were also observed in InGaAs layers grown on (110) InP substrates by MBE [10]. Jen et al. [11] have reported the formation of chalcopyrite-type ordered structure in GaAsSb layers grown by OMVPE on (001) InP substrates. Shahid et al. [12] initially observed CuPt-type ordering in InGaAs layers deposited on (001) InP substrates by VPE. Subsequently, a number of investigators have seen this structure in InAlAs, GaAsSb, GaInP and InGaAsP layers [12-25].

Of the four possible {111} CuPt-type ordered variants, only two are seen [12-25]. Depending upon the composition of a ternary layer, ordering is observed either on $\{111\}_V$ planes or on $(\bar{1}\bar{1}\bar{1})_{III}$ planes. For example, if the layer composition is ABC_2 where A and B atoms belong to group III, ordering involves group V atoms centered tetrahedra. These tetrahedra define the $(\bar{1}\bar{1}\bar{1})_V$ planes of the zinc-blende lattice. On the other hand, if the layer composition is A_2BC, where B and C atoms belong to group V, group III atoms centered tetrahedra are involved in ordering, i.e., ordering occurs on $\{111\}_{III}$ planes. This is borne out by the recent work of Bellon et al. [26].

The situation regarding the choice of the {111} ordering plane in the quaternary layers is more complicated. Shahid and Mahajan [22] have shown experimentally that the CuPt-type ordering in InGaAsP layers occurs on the $\{111\}_{III}$ planes. This choice must be determined by the difference in the driving forces for ordering for the two sets of atoms on their respective sub-lattices.

In order to develop a further understanding of the evolution of fine scale microstructure in ternary and quaternary epitaxial layers, we have investigated the orientation dependence of such microstructures resulting from phase separation in InGaAsP epitaxial layers grown by LPE. In addition, we have evaluated the influence of growth technique on phase separation, ordering and effects of growth temperature and rate on ordering. Results of these studies constitute the present paper.

PHASE SEPARATION

To assess whether or not the two-dimensional decomposition observed in InGaAsP layers grown on (001) InP substrates is generic in nature, we have deposited InGaAsP epitaxial layers on (110), $(111)_{In}$ and (123) InP substrates by LPE. The data shows that the decomposition is indeed two-dimensional in nature and is absent along the growth direction in all cases. Furthermore, it appears that phase separation cannot be suppressed using MBE, and growth temperature affects the periodicity of phase separated microstructures.

Shown in Fig. 1 are a series of dark-field TEM micrographs obtained from an InGaAsP layer grown on a (001) InP substrate by LPE; the emission wavelength of the layer is 1.3 μm. These

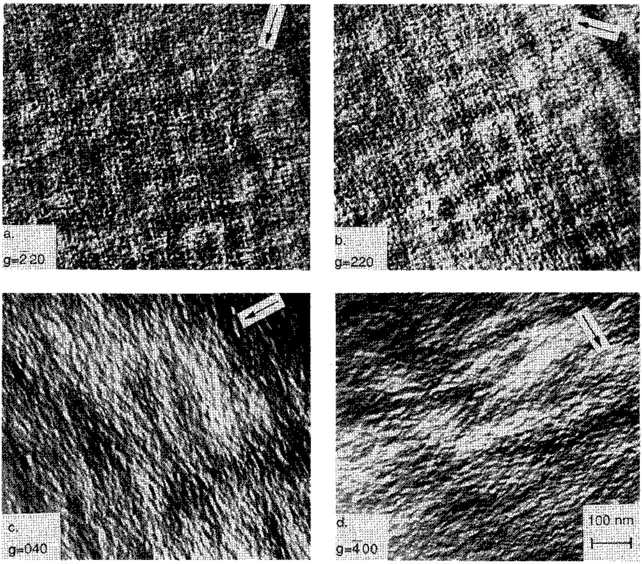

Figure 1. Dark-field electron micrographs obtained from an InGaAsP layer grown by LPE on a (001) InP substrate; emission wavelength is 1.3 μm. Operating reflections in (a), (b), (c) and (d) are, respectively, $\bar{2}20$, 220, 040 and $\bar{4}00$. Arrow in each micrograph delineate a <100> direction lying in the (001) plane.

micrographs show both the fine scale and the coarse contrast modulations. From the results presented in Fig. 1, it can be ascertained that: (i) both types of modulations lie along the [100] and [010] directions, and (ii) the principal strains associated with these modulations are parallel to the directions of modulations, and the periodicity of the fine scale speckle contrast is approximately 8nm.

The results obtained when the same layer is examined in a (110) cross-section, are shown in Fig. 2. In Fig. 2(a), the layer is imaged in dark-field with the 220 reflection which is perpendicular to the growth direction. The fine scale modulations are clearly visible. The same area is imaged with the 004 reflection which is parallel to the growth direction. Two interesting features are noted from Fig. 2(b). First, the fine scale contrast modulations seen in Fig. 2(a) lying normal to the growth direction are out of contrast for this reflection. This indicates that their principal strain components lie in the substrate surface. Second, contrast modulations are not observed along the growth direction, an observation consistent with the earlier works of Chu et al. [7] and Norman and Booker [8].

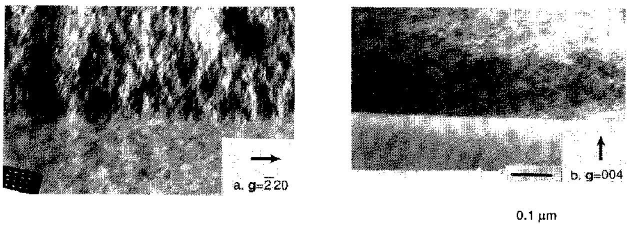

Figure 2. Dark-field electron micrographs obtained from a cross-section of the film shown in Fig. 1. Operating reflections in (a) and (b) are 220 and 004, respectively. These images demonstrate the absence of phase separation along the [001] growth direction. Marker represents 50nm.

A plan-view image obtained from an LPE grown (001) InGaAsP film emitting at 1.2 μm is shown in Fig. 3. The microstructural features of this film and their behavior under different diffraction conditions are much the same as that of the film shown in Fig. 1, with the exception that only one variant of coarse modulations is observed.

Figure 4 shows a series of dark-field micrographs using reflections that lie in the growth plane obtained from an InGaAsP epitaxial layer grown on a $(111)_{In}$ substrate by LPE. A fine speckle contrast similar to that observed in the (001) layer, Fig. 1, can be seen in all the micrographs. The coarse modulations can also be seen in Figs. 4(a) and (b).

In order to assess the alignment of the fine scale structure, laser diffraction studies were carried out. The patterns, obtained from the negatives used to print the micrographs, are shown in the upper left corner of each of the micrographs in Fig. 4. Each pattern shows streaking or diffuse intensity which is characteristic of diffraction from periodic modulated microstructures. However, in each case the elongation is parallel to the operating reflection. It appears that the laser diffraction is occurring from the randomly oriented modulations that are favorably aligned with the operating reflection such that they provide strong contrast. The contrast from modulations that are oriented normal to the operating reflection is much stronger in Fig. 4 and the incident laser beam is diffracted preferentially from these modulations. It is therefore concluded that the fine scale modulations in Fig. 4 are periodic, but not crystallographically aligned.

When the layer in Fig. 4 is examined in cross-section using the 222 reflection, fine scale speckle contrast is again not observed. This implies that phase separation in this case is also two-dimensional in nature and decomposition does not occur along the growth direction.

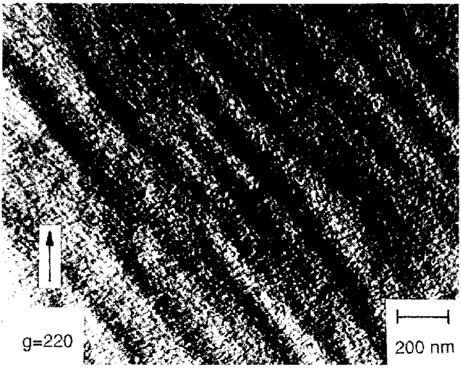

Figure 3. Dark-field electron micrographs obtained from an LPE grown InGaAsP emitting at 1.2 μm. Operating reflection is 220. Note that only the [100] coarse contrast modulations are present.

In Fig. 5, dark-field images from an InGaAsP LPE layer grown on (110) InP substrates are shown. It is evident from Fig. 5(a) taken with $\vec{g} = 2\bar{2}2$ that the fine scale speckle contrast is present. It can be ascertained from Figs. 5(b) and (c) that the overall microstructure is composed of two orthogonal modulations along the [001] and [1$\bar{1}$0] directions and that the principal strain components associated with the modulations are parallel to the directions of the modulations. Further, the respective periods of modulations along the [001] and [1$\bar{1}$0] directions are 6 and 5nm. It is emphasized that decomposition along the growth direction does not also occur in the (110) films.

Figure 6 shows dark-field images obtained from an InGaAsP film grown on a (123) InP substrate. This orientation was chosen because it only contains the [111] direction that is elastically harder that <100> and <110> directions. From these micrographs, it can be seen that the microstructure consists of fine scale modulations along the [0$\bar{3}$2] and [$\bar{3}$01] directions and that the strains are parallel to the modulation directions. In addition, decomposition is not observed along the [123] direction, a result consistent with the observations on (001), $(111)_{In}$ and (110) InGaAsP epitaxial layers.

In order to understand the observed preference of modulation directions for various substrate orientations, McDevitt [27] has calculated elastic work associated with the occurrence of composition modulations along various directions in the (001), (110), (111) and (123) planes. The polar plots of the relative values of elastic work that is associated with creating composition modulations in the substrate plane are shown as Fig. 7. It is apparent from this figure that modulations evolve along those directions in the substrate surface which minimize the coherent strain energy of the

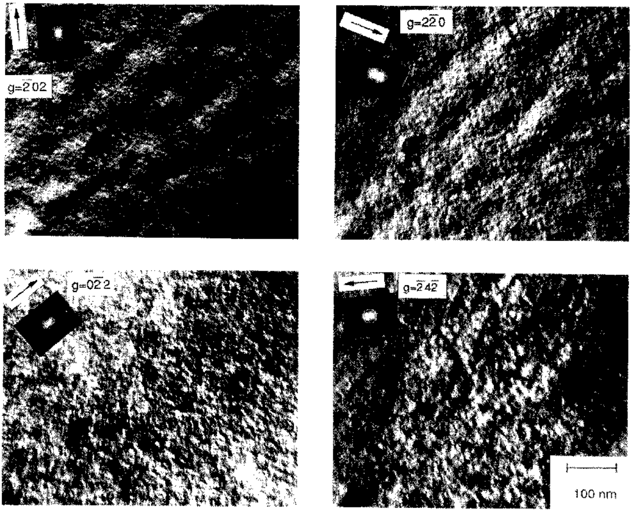

Figure 4. Dark-field micrographs obtained from an InGaAsP layer deposited by LPE on a$(111)_{In}$ InP substrate. Laser diffraction patterns in upper left corner of each micrograph demonstrate the presence of periodicity but lack of crystallographic alignment.

transformation. The observation that the alignment of modulations is determined by the anisotropy in the substrate surface, rather than bulk anisotropy, is consistent with the assessment that phase separation occurs at the surface while the layer is growing.

Several investigators have shown that phase separation also occurs in layers grown by VPE [7,12,22], OMVPE [24] and MBE [8]. We have extended these studies to InGaAs films grown by MBE (001) InP substrates. Shown in Fig. 8 are dark-field images obtained from such a sample. The wavelength of the speckle microstructure is finer than that observed in the LPE specimens, Fig. 1. It is difficult to discern the alignment of modulations for the $\bar{2}20$ and $2\bar{2}0$ reflections, Figs. 8(a) and (b). However, when the 400 type reflections are used, Figs. 8(c) and (d), the speckle microstructure appears to be composed of two orthogonal waves which lie along the two in-plane <100> directions. Further, just like in the LPE case the modulations have principal strain components along the <100> directions. The period of the modulations is ~ 5-6nm as ascertained from Figs. 8(c) and (d).

In order to assess the effects of growth parameters and composition on the modulation wavelength, measurements were made on (001) films grown by three different methods. These results are summarized in Table 1.

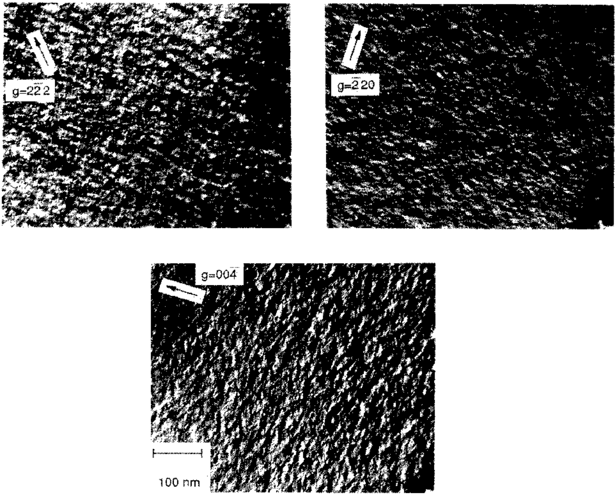

Figure 5. Dark-field micrographs obtained from an InGaAsP layer deposited by LPE on a (110) InP substrate; emission wavelength is 1.3 μm. Speckle contrast appears to be composed of two orthogonal modulations along the [001] and [$\bar{1}$10] directions.

Table 1. Shows Dependence of Periodicity of Modulations on Growth Technique, Growth Temperature and Composition of $In_{1-x}Ga_xAs_yP_{1-y}$ Layers.

Growth Technique	Growth Technique °C	Emission Wavelength (μm)	Composition		Period of Modulations (nm)
			x	y	
LPE	595	1.25	0.2	0.42	8.3
LPE	595	1.15	0.13	0.28	8.7
LPE	595	1.33	0.25	0.54	8.7
OMVPE	625	1.04	0.05	0.12	7.5
OMVPE	625	1.65	0.47	1.00	8.1
MBE	500	1.65	0.47	1.00	7.0
MBE	450	1.65	0.47	1.00	3.5
MBE	400	1.65	0.47	1.00	3.0

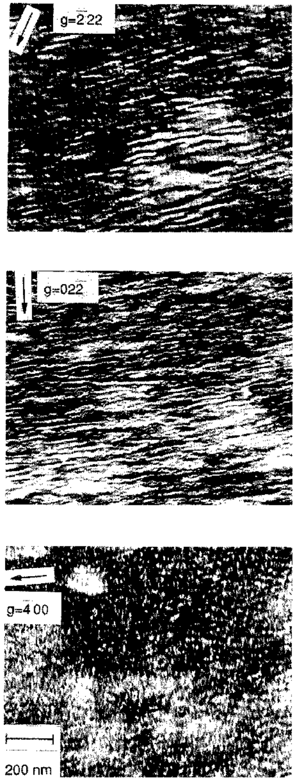

Figure 6. Dark-field electron micrographs obtained from an InGaAsP layer grown by LPE on a (123) InP substrate.

The following trends are evident from Table 1. First, for a given growth temperature and a growth technique, the period of modulations is independent of the composition of the layer, a result consistent with the earlier work of Mahajan et al. [6]. Second, comparing the LPE and OMVPE results it appears that the surface mobility of atoms in LPE is slightly higher than that in OMVPE because the wavelength is larger in the case of LPE. Third, for a given growth technique and composition, the growth temperature has a very strong effect on the period of modulations.

Several significant results emerge from this work: (i) phase separation is two-dimensional in nature and does not occur along the growth direction for the four orientations examined; (ii) coarse contrast modulations are sometimes observed only along one of the two <100> directions lying in the (001) growth plane; (iii) the periodicity of modulations appears to depend on the orientation of the

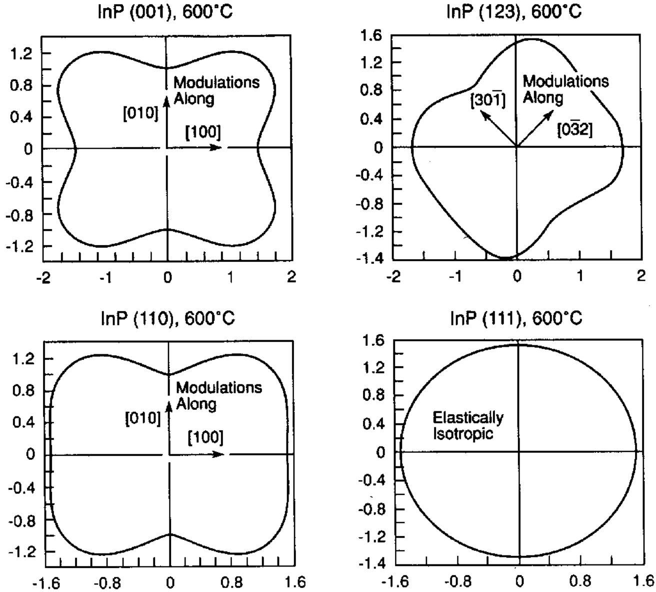

Figure 7. Polar plots of the relative values of elastic work that is associated with the creation of composition modulations in the (001), (110), $(111)_{In}$ and (123) planes of InP.

underlying substrate; (iv) phase separation is observed in layers grown by techniques where growth is controlled by kinetic factors, and (v) for a given growth technique and growth temperature, the period of modulations is independent of the layer composition.

The above results are internally consistent if it is assumed that the speckle structure evolves by phase separation occurring at the surface during growth. If the fine scale structure were to develop by phase separation occurring in the bulk after the layer growth as suggested by Norman and Booker [8], decomposition along the growth direction should dominate, because the layer is thin in that direction, and therefore it is easier to accommodate transformation-induced strains along that direction. This, however, is not observed in this work or in previous studies [7,24]. Accepting that phase separation indeed occurs at the surface while the layer is growing, then the periodicity of the modulations would be determined by the surface mobility of atoms consituting a layer. This, in turn, would depend on the orientation of the underlying substrate, the growth temperature and the growth technique. Further, the effect of layer composition on the period of the modulations may not be significant, but it would affect the amplitude. The preceding assessment is consistent with the results of this study.

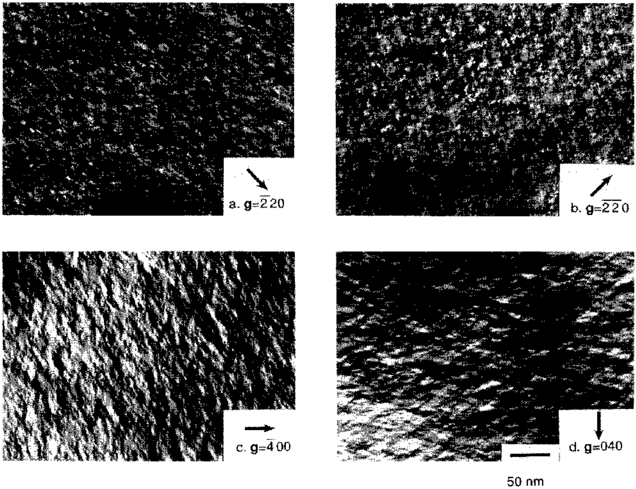

Figure 8. Dark-field images obtained from an InGaAs layer grown by MBE on a (001) InP substrate. Comparing these results with those shown in Fig. 1 it is evident that the modulations in the MBE film have strain fields identical to those of the LPE film, Fig. 1.

Regarding the coarse contrast modulations, it is proposed that they are an artifact of thin samples. Two-dimensional strains associated with the fine scale structure could very well be accommodated by periodic buckling of a thin film as suggested by Alerhand et al. [28]. It is envisaged that after a layer has been thinned, it undergoes buckling. The occurrence of buckling depends in turn on the thickness of the layer: thinner regions may not buckle because strains are not large, while strains may not be sufficient to buckle thicker regions. This suggestion is borne out by the results of Treacy et al. [29] on the thickness dependence of coarse contrast modulations.

ATOMIC ORDERING

To highlight the status of current understanding of atomic ordering in ternary and quaternary epitaxial layers of III-V compound semiconductors, two features will be presented: (i) characteristics of CuPt-type ordering, and (ii) influence of growth rate and growth temperature on CuPt-type ordering.

Shown in Fig. 9(a) is a <110> cross-section of a double heterostructure consisting of InP/InGaAsP/InP layers that was grown by VPE on (001) InP substrates. The diffraction pattern observed from the InGaAsP layer is shown in Fig. 9(b). Superlattice spots lying half-way between the <111> matrix spots are seen. When the second <110> section was examined, superlattice reflections were not observed implying that ordering is occurring only on two of the four {111} variants. It has been ascertained experimentally that ordering occurs on the $\{111\}_A$ planes in the quaternary layer. As first argued by Shahid et al. [12], the above observation can be explained in terms of CuPt-type ordering. Taking the case of $In_{0.53}Ga_{0.47}As$ as an example, they have suggested

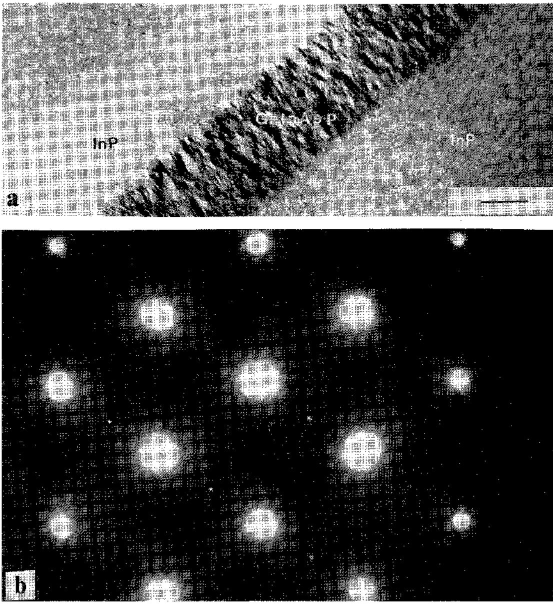

Figure 9. (a) Electron micrograph showing a $<1\bar{1}0>$ edge-on view of a heterostructure consisting of InP/InGaAsP/InP layers grown by VPE on a (001) InP substrate. (b) A $\{1\bar{1}0\}$ electron diffraction pattern obtained from the InGaAsP layer shown in (a). Note the presence of superlattice reflections. Marker in (a) represents 0.1 μm.

that the observed doubling of periodicity along the <111> can be understood if it is assumed that A(III) a(V) B(III) b(V) C(III) c(V).... in the random alloy changes to A(In-rich) a(V) B(Ga-rich) b(V) C(In-rich) c(V) A(Ga-rich) a(V) b(In-rich) b(V) C(Ga-rich) C(V) A(In-rich).... in the ordered alloy.

A number of investigators have seen this type of ordered structures in $AlInAs_2$ [13], Ga_2AsSb [14,15] and $GaInP_2$ [16-21] layers. In addition, Jen et al. [11] have reported the existence of CuAu-I type and chalcopyrite structures in Ga_2AsSb layers. The latter is a surprising result, because unless the three ordering energies are equal, the three different types of ordered structures, i.e., CuAu-I, chalcopyrite and CuPt-type, could not coexist! The resolution of this dilemma may lie in the suggestion of Shahid and Mahajan [22] who have argued that the co-existence of two CuPt-type variants could locally produce atomic arrangements which resemble CuAu-I type and chalcopyrite structures.

The growth temperature and growth rate have a considerable influence on the size of domains. This effect is illustrated in Fig. 10. Figure 10(a) shows domains in $(Ga,Al)InP_2$ layers grown on (001) GaAs substrates by OMVPE at a low growth temperature (650°C) but a high growth rate, and Fig.

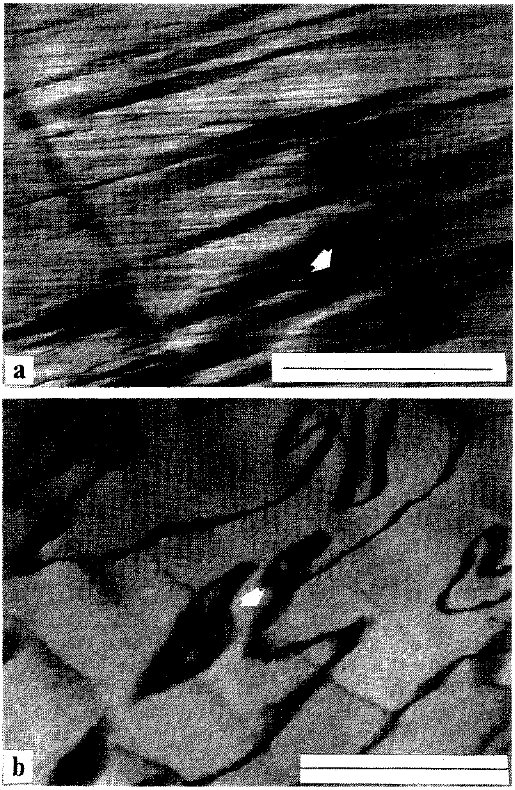

Figure 10. Domain boundaries observed in (Ga,Al)InP_2 layers grown at different rates on (001) GaAs substrates by OMVPE: (a) high, and (b) low growth rate. Reflections used for forming images in (a) and (b) are, respectively, 111 and $3\bar{1}1$ superlattice spots. Markers in (a) and (b) represent 0.2 and 0.5 μm, respectively.

10(b) exhibits domains seen in (Ga,Al)InP_2 layers grown at a high growth temperature (675°C) and a low growth rate.

The formation of domains can be rationalized by referring to Fig. 11. In Fig. 11 surfaces AB and CD delineate a step of height $\frac{a}{4}$ at the (001) GaAs surface. These two surfaces are distinctly different: surface AB is occupied by the As atoms, whereas Ga atoms define CD surface. Now imagine that the stepped surface is subjected to a flux of In, Ga and P atoms - the atoms required to grow a layer of $GaInP_2$. It may be argued following Suzuki et al. [31] that As atoms along the row B would bond to a row of Ga atoms, position G in Fig. 11, and the rows of In and Ga atoms would

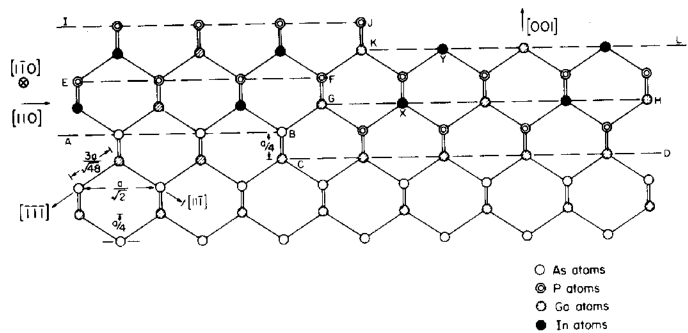

Figure 11. Schematic illustrating the role of a surface step in ordering and in forming domain boundaries in a $GaInP_2$ layer grown on a (001) GaAs substrates.

alternate as shown. The crucial point is which atoms do occupy the row marked X in Fig. 11. If this row is occupied by the In atoms as shown, then the alternating arrangement of the two types of atomic rows would be commensurate with each other on either side of the step. However, if the row is occupied by the Ga atoms, then a volume of Ga(4)P tetrahedral units is produced. As a result of the atomic attachment, the layer will thicken and the initial step will assume the position EFGH. Invoking arguments for atomic attachment at positions K and Y similar to those used for positions G and X, it can be seen that two distinct situations develop: (i) the ordered regions on either side of the step are commensurate with each other, and (ii) the ordered regions are separated from each other by the tubes of the binary material. The position of these tubes shift laterally during growth. It is suggested that these tubes are responsible for the domain contrast observed in Fig. 11. This assessment is consistent with the observations of Eugarde et al. [26] on the evolution of domains in $GaInP_2$ layers from steps present on the GaAs substrate.

It is apparent from the above discussion that ordering, like phase separation, occurs at the surface while the layer is growing, i.e., the two occur concomitantly. The crucial question is how would the evolution of In-rich and Ga-rich regions due to surface phase separation affect CuPt-type ordering? The answer is that it should not because CuPt-type ordering is observed in $Ga_{1-x}In_xP$ layers even when X deviates substantially from 0.5 [31].

Srivastava et al. [32] have predicted that in the bulk chalcopyrite ordered structure is stabler than CuAu-I and CuPt-type structures. This result is not consistent with the observations discussed above. The recent work of Zunger and co-workers [33] has shown that if ordering is assumed to occur at the surface as discussed above and reconstruction of the (001) surface is taken into consideration, then CuPt-type ordered structure has the lowest energy. It is thus now possible to reconcile theory with experiment.

With the exception of a single observation where long range order has been observed in layers grown by LPE [34], ordering is not observed in LPE grown layers. This may be due to the fact that the presence of liquid on the growth surface prevents its reconstruction.

SUMMARY

Phase separation in InGaAsP layers grown on (001), (110), $(111)_{In}$ and (123) InP substrates by LPE is two-dimensional in nature, and does not occur along the growth direction. These results taken together is observed along the soft directions lying in the growth plane. Taken together these results very strongly suggest that phase separation indeed takes place at the growth surface while the layer is growing.

The evolution of wavelengths during surface phase separation is determined by surface mobility of atoms constituting an epitaxial layer. This in turn depends on the growth temperature, orientation of the underlying substrate and growth technique. Amplitude of the modulation is the only parameter depending on layer composition.

CuPt-type ordering is observed in InGaAsP layers grown on (001) InP substrates by VPE. Out of the possible four {111} ordered variants, only the two that lie on $\{111\}_{In}$ planes are seen. The observed doubling of periodicity along the <111> directions in $InGaAs_2$ results from the following arrangement of the {111} planes: A(In-rich) a(V) B(Ga-rich) b(V) C(In-rich) c(V) A(Ga-rich) a(V) B(In-rich) b(V) C(Ga-rich) c(V) A(In-rich).....

Growth temperature and growth rate effects on domain size in $(Ga,Al)InP_2$ layers grown on (001) GaAs substrates by OMVPE are observed by the generation of large domains at high growth temperature and low growth rate. A model has been proposed for the formation of domains and involves their nucleation from surface steps. Like phase separation, ordering occurs on the surface while the layer is growing and very likely involves reconstruction of the (001) surface. From these results it is inferred that phase separation and ordering occur concomitantly at the surface while the layer is growing.

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