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Interfacial roughening in lattice-matched GaInP/GaAs heterostructures

Y.Q. Wang[a], Z.L. Wang[a,*], T. Brown[b], A. Brown[b], G. May[b]

[a]*School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA 30332-0245, USA*
[b]*Department of Electrical Engineering, Georgia Institute of Technology, Atlanta, GA 30332, USA*

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Abstract

The microstructure of solid source molecular beam epitaxy (MBE) lattice-matched GaInP-GaAs heterostructures has been studied by transmission electron microscopy (TEM). It is shown that atomic-scale roughening occurs in the first several (∼five) interfaces, and beyond which roughening is developed into micrometer-scale. The {113} faceted roughening occurs in the GaInP-on-GaAs interfaces, leading to the formation of V-shaped grooves. Dislocation dipoles are observed in the top, facet surface and bottom regions of the grooves, and there is no obvious difference. For GaInP layers exhibiting atomic-scale roughening, a small number of dislocations, mostly 60°-type and in a dipole configuration, are confined in a zone of approximately 5.0 nm in width along the interface, and dislocations are rare deep inside the layers. For GaInP layers, in contrast, there is a high density of dislocations in both the regions, and it has remarkable interfacial roughening. Based on their distribution and configurations, the dislocations are believed to result from the compositional modulation occurred in the GaInP layers. The interplay of roughening, dislocations and compositional modulation has been discussed in the light of the morphological features.

Keywords: Interfacial roughening; Heterostructure; GaInP-GaAs; Molecular beam epitaxy

1. Introduction

Interfacial abruptness and roughness of semiconductor heterostructures are two key microstructural factors in determining quantum effects in low dimensional structures, especially quantum wells. Non-abrupt interface results in a shallow and non-rectangular well and causes a shift in exciton energy levels towards higher energies. Roughness of the interface causes fluctuation in the energy level, which in turn leads to reduction of carrier mobility due to strong scattering and broadening of the photoluminescence [1]. Growing abrupt and smooth interfaces is, therefore, critical for device technology. However, epitaxial growth, which is the most common technique for growing heterostructures, is essentially an interfacial engineering process and the epilayers are signatures of what has happened at the final stage of this kinetically-controlled process. Interface smoothness is ultimately determined by that of the growing interface. Therefore, insight into the interfacial structure and morphology of the semiconductor heterostructures promises an important step toward a deep understanding in the atomistic epitaxial growth mechanism and the rate-controlling factors for the growth of morphologically and compositionally sharp interfaces.

In our recent work on lattice matched GaInP/GaAs heterostructures, we have identified a special misfit dislocation configuration — dislocation dipoles, formed in the GaInP layer along the GaInP-on-GaAs interface [2]. All of the GaInP epilayers have been found to undergo lateral compositional modulation with irregular spacing and a distinguished morphology [3]. Besides these two interesting phenomena, considerable interfacial roughening has been observed to occur after growing a certain number of GaInP and GaAs layers. In this paper, we will present a detailed description of this

* Corresponding author. Tel.: +1-404-894-8008; fax: +1-404-894-9140.
E-mail address: zhong.wang@mse.gatech.edu (Z.L. Wang).

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Table 1
Experimental factors and settings

1	As species	As_2, As_4
2	Layer configuration	Normal, Inverted
3	Substrate temperature	475–520°C
4	QW interrupt time	10–60 s
5	As BEP	$2e^{-6}$ torr, $6e^{-6}$ torr
6	P_2 BEP	$2e^{-6}$ torr, $6e^{-6}$ torr

interfacial roughening phenomenon in terms of microstructural evolution. We first present our microstructural observation of the heterostructures. Then, a systematic discussion will be given about the possible mechanisms involved in the roughening process.

2. Experimental

Undoped GaInP-GaAs multiple quantum well (QW) structures were grown on a GaAs(001) substrate using solid source molecular beam epitaxy (MBE). The input factors of interest were substrate temperature (475°C for GaAs and 520°C GaInP layer growth), beam equivalent pressure (BEP) of the As source, As species, BEP of the P source and QW interrupt time. The input factor settings are given in Table 1. Each period of the QW structure consisted of a 400-Å buffer layer and a heterointerface of 50 Å GaInP and 50 Å GaAs (see Fig. 1). The GaInP/GaAs heterointerface was grown in both its normal and inverted layer configurations.

It should be noted that these samples were grown as a part of a statistical experiment design aiming to understand the anion exchange at the interface. We were able to produce samples with good interface. However, we did observe interfacial instabilities in some of these structures under certain growth conditions. In this paper, we will just discuss the most typical microstructure of these imperfect samples.

[110]/[110] cross-sectional specimens for transmission electron microscopy (TEM) were prepared by gluing two growth surfaces together with M-610 crystal bond. The specimens were bonded in such a way that one-half was along the [110] orientation and the other along the [110] orientation. Therefore, the heterostructure could be examined simultaneously from orientations. All of the specimens were polished mechanically, dimpled and ion milled at 5 kV using Ar^+ ion beam until perforation. The milling angle was 15° and the specimen temperature was kept at 100 K. The specimens were examined at 400 kV using JEOL 4000EX microscope. In our samples, no obvious microstructural difference was observed between [110] and [110], so for clarity, we simply use [110] to refer to the [110]/[110] cases and make no attempt to differentiate them.

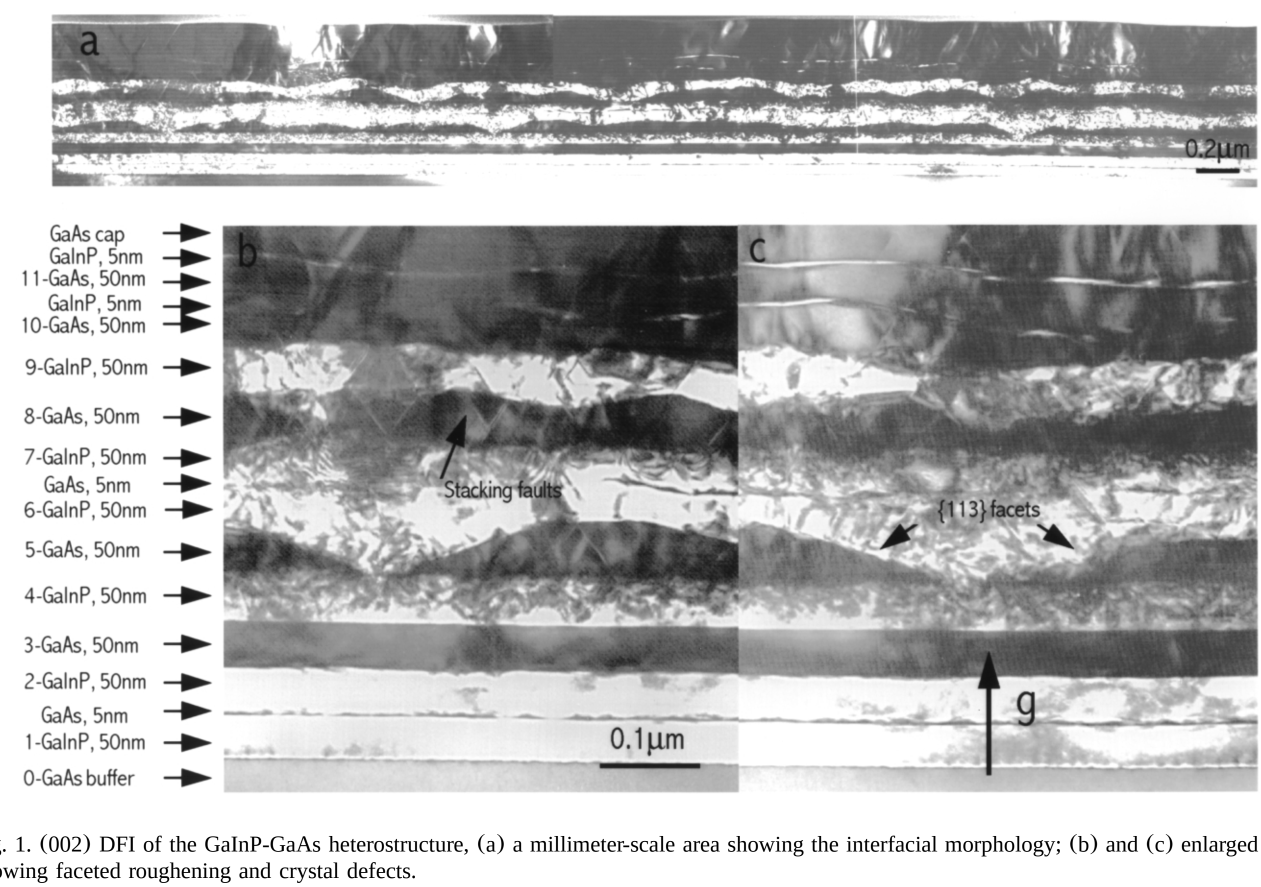

Fig. 1. (002) DFI of the GaInP-GaAs heterostructure, (a) a millimeter-scale area showing the interfacial morphology; (b) and (c) enlarged areas showing faceted roughening and crystal defects.

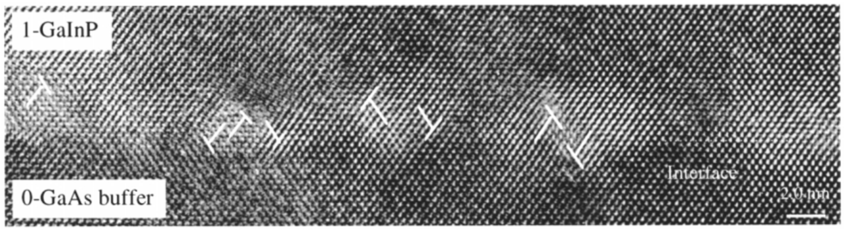

Fig. 2. HR-TEM image showing misfit dislocation dipoles along the interface between the GaAs buffer and the first GaInP overgrowth.

3. Results

Fig. 1 shows [110] cross-section dark field images (DFIs) of the heterostructure formed by (002) reflection. Due to the distinguished structure scattering factor between GaInP and GaAs, the GaInP and GaAs epilayers are readily differentiated in the (002) DFI. The GaInP layers are imaged as brighter bands, while the GaAs layers are imaged as dark gray bands. As shown in Fig. 1, both the GaInP-on-GaAs (GaInP/GaAs) and GaAs-on-GaInP (GaAs/GaInP) interfaces appear well defined on the micro-scale for the first several layers. Apparent interfacial irregularities set in from the growth front of the fourth GaInP barrier layer (4-GaInP). As a result, the 5-GaAs/4-GaInP interface appears as a roughened interface instead of a smooth one. The subsequent GaAs barrier layer exhibits a much rougher growth front, as shown in the typified enlarged regions. In some parts, well-developed V-shaped grooves are formed on the 6-GaInP/5GaAs interface. The facets are found to be close to {113} planes. Generally, roughening of the GaInP/GaAs interfaces is much more remarkable than the GaAs/GaInP interfaces. It seems that the GaInP layer grows faster in the V-shaped groove, thereby producing some recovery of the smooth (001) growth plane during its growth. However, this smoothening effect can also be seen in the growth of the thick GaAs cap layer.

Crystal defects, such as stacking faults and threading dislocations, were rarely observed in the first several epilayers. This seems to match the good planarity of the corresponding interfaces. However, from the 4-GaInP layer, where apparent roughening starts (see Fig. 1), the crystallinity seems to degrade considerably due to the large number of crystal defects. The planar defects are readily identified as stacking faults, which typically form the so-called Lomer–Cotrell lock. In roughened strained films, such defects were usually found to form in the bottom regions of troughs or grooves, where high stress concentration is located [4]. In our sample, it seems difficult to justify such a correlation. Due to the presence of local strain field associated with the compositional modulation occurred in GaInP epilayers [3], and the fact that the 60° dislocations are at nearly edge-on position, the dislocations are not so readily recognized as the stacking faults. However, the edge-on dislocations will be readily revealed by high resolution TEM images, as shown in the following sections. The above observations clearly indicate that an introduction of the crystal defects is somehow interrelated with the roughening phenomenon. Such a high density of crystal defects is apparently out of one’s expectation from a lattice-matched heterostructure grown on an essentially dislocation-free substrate.

Fig. 2 shows a high resolution TEM image of the interface between the GaAs buffer and the 1-GaInP barrier layer. The ‘$\perp$’ symbol represents a dislocation running along [110] or the electron beam direction. It can either be a 60° dislocation or a Lomer dislocation. As shown in Fig. 2, there exist misfit dislocations and lattice distortions on the side of 1-GaInP layer along the interface, although the lattice mismatch is negligible. These misfit dislocations were found to form a dipole configuration with two dipole dislocations being separated by approximately 3.5 nm. The spacing between the two dislocation dipoles is irregular. Lengths of up to several tens of nanometers were often found dislocation free. A detailed description has been given elsewhere [2]. It should be noted that although the interfaces appear smooth and well defined at micro-scale, they are rough at the atomic level. However, a quantitative description of the roughness is difficult because the exact position of the interface can be hardly determined due to the influence of the local strain field and dislocations at the interface.

Shown in Fig. 3 is a typical HR-TEM image of the 1-GaInP and 2-GaInP epilayers with a 5.0-nm-thick GaAs layer sandwiched in between. It is found that the density of dislocation dipoles along the GaInP-on-GaAs interface is higher than along the GaAs-on-GaInP interface but is lower than along the GaInP-on-GaAs buffer interface. Furthermore, the GaInP-on-GaAs interface is rough as compared with the GaAs-on-GaInP interface. Similar interfacial morphology was observed between the 2-GaInP and 3-GaAs and that between the 3-GaAs and 4-GaInP. Although a certain number of misfit dislocations, mostly in a dipole configuration, are present along the interface, they were much less frequently observed inside the first and second GaInP barrier layers away from the interface. However, considerable lattice

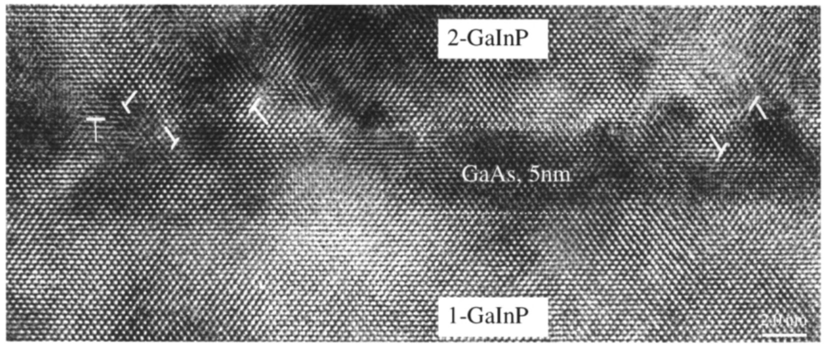

Fig. 3. HR-TEM image showing the first GaInP (1-GaInP) and the second GaInP (2-GaInP) barrier layers with a 5.0-nm-thick GaAs layer sandwiched in between. Note the misfit dislocation dipoles and the interfacial roughness.

distortions are observed along the growth direction. These distortions tend to modulate laterally with a very irregular spacing. This distortion modulation, manifested as a contrast modulation, is believed to be related to the lateral compositional modulation occurred inside the GaInP epilayers [3].

As indicated in Fig. 1, apparent roughening commences from the fourth GaInP barrier layer. A typical HRT-EM image of the 5-GaAs/4-GaInP interface and its adjacent regions is shown in Fig. 4. It can be seen that the density of dislocations, mostly of 60° type, is considerably increased along the GaAs-on-GaInP interface. Unlike the first several epilayers where the small number of dislocations are usually confined in a zone of approximately 5∼10 nm wide, 60° dislocations in the 4-GaInP layer can be frequently observed in regions far away from the 5-GaAs/4-GaInP interface. More interestingly, these dislocations seem to present a similar dipole configuration, suggesting that they may have the same formation mechanism as those formed along the interface. However, they tend to distribute vertically or along the growth direction, rather than laterally. A careful observation also shows that these dislocations were formed in the highly lattice-distorted regions, which are roughly parallel to the growth direction. As we reported earlier [3], the lattice-distortion was caused by compositional modulation. The presence of dislocations in these regions indicates that their formation is intimately related with the compositional modulation process. Presumably, they are introduced to help relieve the strain energy associated with a coherent spinodal. It is also found that the dislocation density tends to increase inside the GaInP layer from the 4-GaInP/3-GaAs interface to 5-GaAs/4-GaInP interface. This leads us to believe that the dislocations are generated due to the compositional modulation in the GaInP layer. Another piece of evidence in favor of this argument is that dislocations are rarely observed in the GaAs layer, in

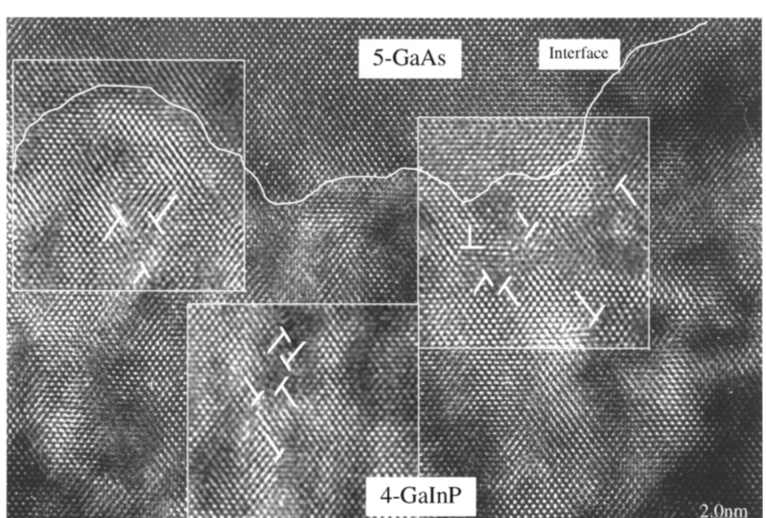

Fig. 4. HR-TEM image showing the rough interface between the fifth GaAs (5-GaAs) and the fourth GaInP (4-GaInP) layers and its adjacent areas. The framed areas are FFT filtered.

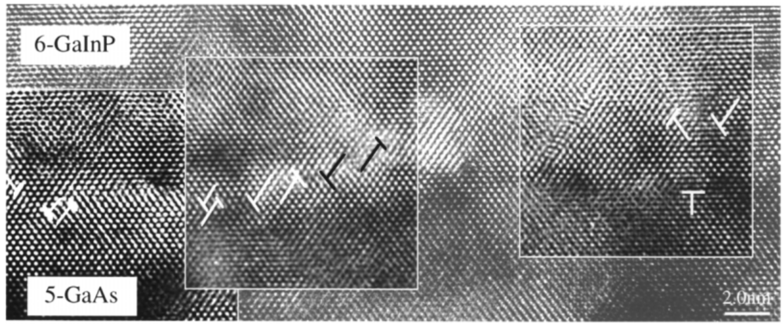

Fig. 5. HR-TEM image, taken from the peak region of a ripple, showing the interface between the sixth GaInP (6-GaInP) and the fifth GaAs (5-GaAs) layers. The framed areas are FFT filtered.

spite of the great number of dislocations in the GaInP layer.

Fig. 5 shows an HRTEM image of the 6-GaInP/5-GaAs interface, where, as indicated in Fig. 1, remarkable facet roughening occurs. The micrograph is taken from the peak region of a ripple. Clearly, the interface is heavily decorated with misfit dislocation dipoles and lattice distortions. However, unlike the 4-GaInP barrier layer, the 6-GaInP barrier layer contains far less dislocations and far less severe lattice distortions in regions deep into the layer. The faceting regions were found to exhibit similar rough interfacial morphologies, although they can be defined as {113} plane microscopically.

4. Discussions

4.1. Interface roughening

Interfacial roughening in epitaxial growth can be roughly categorized into intrinsic roughening and kinetic roughening. Intrinsic roughening refers to a phenomenon in which the interface roughening is favored thermodynamically and hence is a spontaneous process. In the recent effort in engineering nanostructures, there is an increasing trend to grow strained heterostructures to satisfy the wide spectrum of engineering needs for fabricating novel electronic/optoelectronic devices. This effort is driven to face the challenge that nature has offered only a few limited choices in selecting pairs of different semiconductor crystals that have the same crystal structure, are lattice-matched, and are dissimilar enough in their electronic and optical properties to make heteroepitaxy useful. However, straining of the epilayers adds more flexibility in tailoring the electronic/optoelectronic properties in a way that might be impossible otherwise. The practice of strained heteroepitaxy has proved to be a great challenge. One of the most frequently encountered problems is interfacial roughening. Theoretical analysis indicates that a strained epitaxial film with a planar surface is thermodynamically unstable with respect to that with a wavy morphology [5]. Introducing roughness at the interface or surface is an alternative mechanism to misfit dislocations for relieving the strain energy caused by lattice mismatch. This morphological evolution is counterbalanced energetically by the increase in surface energy. Roughening will not occur until the free energy reduction in the system by strain relief outweighs the increase in free energy due to an increase in interface area and step formation. Assume the epitaxial film takes a sinusoidal undulation with a periodicity λ, then it is predicted that the periodicity λ satisfies the inequality: $\frac{t}{\lambda^2} < \frac{E\varepsilon_0^2}{4\gamma\pi^2}$, where t is the undulation amplitude of the epitaxial film, γ is the specific interfacial energy, E is Young's modulus and ε_0 is the misfit strain [6]. The actual morphology also depends on growth conditions, exhibiting a range of configurations, including islands, smoothly undulated surfaces, and grooved or cusped surfaces. The equation explicitly predicts that a planar thin film become more stable when misfit strain tends to zero ($\lambda \to \infty$). This is unnecessarily so kinetically. It has been experimentally shown that under some growth conditions, the interface tends to roughen, resulting in kinetic roughening [7,8]. Contrary to intrinsic roughening, kinetic roughening is a kinetics driven process. At atomic or molecular level, the epitaxial growth process consists of a number of sub-processes: absorption, migration, desorption, reaction, dissociation, incorporation, etc. These kinetic processes determine the interface evolution and are controlled principally by growth temperature, growth rate, interruption time, and the beam equivalent pressure ratio for compound semiconductors. In practical heterosystems, lattice mismatch is almost unavoidable, so both intrinsic and kinetic roughening may occur simultaneously, giving rise to a complex roughening phenomenon. Under certain conditions, intrinsic roughening can be inhibited and hence a planar interface can be kinetically stabilized [9].

Interfacial roughening interacts strongly with formation of dislocations and other kinds of crystal defects.

The formation of undulations in the strained layer produces crests where stress is relieved elastically and troughs where stress is concentrated, irrespective of the sign (positive for tensile and negative for compressive) of the strain in the thin film. Faceting of the undulation sidewalls causes the troughs to develop into grooves or cusps and aggravates the local stress concentration. It has been shown theoretically and experimentally that the stress concentration can greatly assist the formation of misfit dislocations by lowering the large energy barrier to dislocation nucleation in the growing films [4,10]. Controlled annealing of defect-free planar films has shown that surface roughening precedes dislocation formation [11]. It should be emphasized that surface roughening is not a prerequisite for misfit dislocation formation. They are two independent competitive strain relaxation mechanisms. Depending on the growth conditions, relaxation by misfit dislocation may precede interfacial roughening. Lavoie et al. [12] have shown that, in strained InGaAs thin films, generation of misfit dislocations had already occurred before interface roughening, implying the latter is complementary to the former in relieving the strain energy.

The morphological evolution of a growing interface is the result of a balance between roughening and smoothing processes. Stochastic addition or removal of matter tends to roughen the interface, while mass transport by surface energy minimization tends to smooth it. On a roughened interface, surface energy drives mass transport from high curvature to lower curvature regions, from concave-downward regions to concave-upward regions. When a roughened film is strained, there is an additional mass transport driven by strain gradient. The direction of mass flux, however, depends on the sign of strain. Tensile strain causes mass flux in the same direction as that for the interfacial energy driven case, giving rise to a smoothing effect; whereas compressive strain causes an opposite flux, making the interface rougher. This asymmetry may be one of the reasons for the phenomenon observed in Ge/Si(100) systems that films under compression roughening by forming coherent islands while those under tension remain relatively smooth [13]. In the case of compound semiconductor alloy films, an even more interesting result can be observed. Because of the atomic size difference, the constituent atoms interacts with the strain field differently, resulting in a redistribution of atom species; atoms with large atomic size tends to segregate into stretched regions while small atoms tend to segregate in compressed regions. If the undulation and hence the strain field is periodic or quasi-periodic, the composition redistribution will also be correspondingly periodic, leading to the phenomenon of compositional modulation. This coupling effects between morphological and compositional instabilities has been observed experimentally and analyzed theoretically [14,15]. Apparently, if the interfacial energy is strongly composition dependent, one may also observe a compositional modulation phenomenon driven by interfacial energy minimization.

4.2. Facet roughening

Our results clearly show that from the 5-GaAs barrier layer onwards, remarkable {113} facet roughening occurs at the growth front of GaAs layers. Similar results were also reported by Mahalingam and his coworkers [16] in lattice-matched GaInP to GaAs heterostructures grown by gas source MBE. However, as pointed out in the experimental section, we found that the interfacial morphological features appear essentially the same for both [110] and [110] cross-section samples, while Mahalingam et al. reported that the roughening phenomenon was only observed along [110]. They also reported that the V-shaped grooves occurred at almost identical locations on the subsequent GaAs growth front, while no such preference was observed in our sample. Mahalingam et al. argued that the {113} faceting was caused by P_2-on-GaAs interruption. The exposure of (001) GaAs plane to P_2-flux reduced the kinetic barrier and hence changed the (001) growth front to the more energetically favorable the (113) growth front. Their argument implied that roughening of the (001) GaAs growth front was evolved during the interruption. In other words, the (001) growth front was smooth before interruption. If so, the material in the V-shaped grooves, originally GaAs, must be removed by some atomic processes during interruption to form the grooves. Apparently, these processes involved a considerable amount of mass transport.

Assume the diffusivity is on the order of 10^{-13} cm^2 s^{-1} for both Ga and As [17], the diffusion length $[L=(D\tau)^{1/2}]$ can be estimated as approximately 25 nm for $\tau=60$ s (the interrupt time during the growth between the adjacent layers). This value is very close to the half-spacing between the grooves, implying that the mass transport by surface diffusion may be the dominant atomic process in the formation of {113} facets. In our case, however, the spacing ranges at approximately 200 nm, an order higher than the diffusion length, suggesting that facet roughening of the growth front may involve other mass transport mechanism. A possible kinetic process is the desorption of As and incorporation of P into the grooved region. This process will apparently result in some change in the chemical composition about the grooved regions. Further work needs to be done to clarify this possibility.

Facet roughening was also reported by Okada et al. [18] in strained InGaAs on InP heterostructure grown by gas source MBE. In contrast to the results discussed above, they found that faceting of the growth front commenced at the initial growth stage of GaInAs. The strain energy was considered to provide the driving force for the instability of a planar interface with respect to faceting. They further carried out some numerical analysis by using an atomic model where the {113} planes consist of a series of (001) terraces, and locally preserve the (2×4) reconstruction at the (001) surfaces that are separated by double steps. They found that the critical wavelength was orders of magnitude higher than the facet size observed at the earliest stage of growth. This large discrepancy led them to believe that there was another mechanism for the facet roughening other than the Stranski-Krastanov roughening mechanism, i.e. three-dimensional island growth succeeding an initial stage of planar growth. They also conclusively showed that the operating mechanism is sensitive to the strain. The 0.5% tensile-strained film exhibited faceting roughening while the lattice-matched or compressed (-0.5%) growing film was able to maintain a planar growth front. In our sample, the mismatch strain is very small (approx. 0.1%). Therefore the possibility of strain driven roughening is considered small. However, we do not exclude the possibility that high local strain, which is manifested by the severe lattice distortions in the atomic images, may have considerable contribution to the roughening.

4.3. Roughening and compositional modulation

As demonstrated in our results, the first several layer are able to maintain planar and smooth interfaces at micro-scale until the 4-GaInP layer from which apparent roughening commences. We have reported [3] that the GaInP layers underwent lateral compositional modulation. This phenomenon is believed to couple with the roughening process. A rigorous thermodynamic analysis was recently made by Glas [15] on the coupling effect between morphological and compositional instabilities in a stressed alloy with a free surface. It was shown that any alloy is practically unstable with respect to a range of joint surface undulations and compositional modulations. Therefore, the coupling of morphological and compositional instabilities is thermodynamically possible in our sample although the mismatch strain is small. This seems to put us facing a chicken and egg problem: of these two coupling processes, which one occurs first. Lateral compositional modulation causes the interface in compression and expansion alternatively, leading naturally to interfacial roughening. This phenomenon seems to be particularly pronounced in short period superlattices [19]. However, the local strain concentration fields of a roughened surface interact differently with atoms of different sizes, thereby leading to atomic species redistribution or compositional modulation [14]. It seems still premature to reach a conclusion on which one occurs first from our present results. However, interfacial roughening and compositional modulation are not necessarily interdependent. Okada et al. [18] conclusively showed that the GaInAs epilayer lattice-matched to GaP underwent lateral compositional modulation while maintaining a planar growing front.

4.4. Dislocations and compositional modulation

The formation of dislocations in the epilayers results from the lateral compositional modulation occurring in the GaInP layers. This conclusion is drawn from the following facts: (1) there exist dislocations, mostly in a dipole configuration, in regions either along the interface or deep into the GaInP layer; (2) In the 4-GaInP layer, dislocation density increases with the layer thickness. Our large area observation showed that the dislocation density is rather small at the early growth stage of this layer. This can also be inferred from the better planarity of the 4-GaInP/3-GaAs interface; (3) there are relatively negligible number of 60° dislocations inside the GaAs layers despite of a large number of 60° dislocations inside the GaInP layers. As discussed by Wang et al. [3], the introduction of dislocations helps relieve the coherent strain energy so that the spinodal process can proceed in a semicoherent way above the coherent spinodal temperature.

4.5. Roughening and dislocations

Is there any relationship underlying the roughening and formation of dislocations? In a strained film, roughening causes high local stress concentrations, which, in turn, promote the formation of dislocations and stacking faults [4,20]. However, Jonsdottir [21] proposed an alternative model in which formation of misfit dislocations enhances growth rate at the relaxed surface areas and hence causes surface roughening. Experimental evidence supporting this model is the so-called cross-hatch pattern [22]. Our results show that there is a drastic increase in the density of dislocation and stacking faults accompanying the remarkable roughening. However, we found no evidence supporting a defined relationship between the formation of the crystal defects and their locations. In Mahalingam's report [16], no information about dislocations was provided. However, the observation of groove formation at about constant vertical location implies that local strain fields may be present at the grooves. Presently, we tend to think that dislocations considerably influence the roughness through the diffusion kinetic in the case of lattice-matched heterostructure.

5. Conclusions

In the multilayered GaInP-GaAs heterostructure, the first several (five) interfaces roughen at the atomic scale while roughening at the micro-scale is observed in the subsequent interfaces.

The {113} faceted roughening occurs at the GaInP-on-GaAs interfaces, leading to the formation of V-shaped grooves. Dislocation dipoles are observed in the top, facet and bottom regions of the grooves with no obvious difference.

For GaInP layers exhibiting atomic level roughening, a small number of dislocations, mostly 60°-type and in a dipole configuration, are confined in a zone of approximately 5.0 nm wide along the interface. Dislocations are far less frequently observed deep inside the GaInP layers. In contrast, there is a high density of dislocations in both these regions for the GaInP layers that experience remarkable interfacial roughening.

The formation of dislocations is believed to result from compositional modulation occurred in the GaInP layers.

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