

# Crosshatched surface morphology in strained III-V semiconductor films

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The correlation between the surface crosshatched morphology and the interfacial misfit dislocations in strained III-V semiconductor heteroepitaxy has been studied. The surface pattern is clearly seen on samples grown at high temperature (520 °C) and those with small lattice-mismatched ( $f < 2\%$ ) systems. A poorly defined cross hatched morphology was found on layers grown at relatively low temperature (400 °C). As the lattice mismatch of the strained layer becomes larger than 2%, a roughly textured surface morphology is commonly observed in place of actual cross-hatching. Few threading dislocations are observed in the strained layer when the crosshatched pattern develops. It is also noted that the surface crosshatched pattern develops after the majority of the interfacial misfit dislocations are generated. The result suggests that the surface crosshatch pattern is directly related to the generation of interfacial misfit dislocations through glide processes.

## I. INTRODUCTION

Strained heteroepitaxial III-V semiconductors are of great technological importance in electro-optical device applications.<sup>1</sup> One of the commonly observed features of strained heteroepitaxy is the presence of a nonuniform surface morphology. Although the surface morphology of the strained epitaxial layer is generally mirrorlike when observed by regular optical microscopy, a surface crosshatched pattern is revealed when Nomarski interference is applied. This gridlike surface pattern consists of ridges lying along [110] and  $[1\bar{1}0]$  directions when a (001) growth orientation is employed. The presence of the surface relief associated with this crosshatched morphology can be of great significance in device fabrication processes.

The surface crosshatched morphology was first discussed by Burmeister, Pighini, and Greene<sup>2</sup> with regard to lattice-mismatched GaAsP epitaxial layers grown on GaAs. They considered the origin of this structure to be the strain produced by the lattice mismatch. X-ray topography was also applied in a study of surface curvature of the GaAsP/GaAs strained epitaxial system by Kishino, Ogirima, and Kurata.<sup>3</sup> They found that the surface curvature of a wafer in which the crosshatched pattern was clearly visible was always smaller than that of the wafer in which the crosshatched pattern was obscure. The small surface curvature on the crosshatched sample indicates that a greater portion of the strain was relieved by misfit dislocations than in the noncrosshatched sample. Kishino and co-workers concluded that the crosshatched pattern

observed in GaAsP/GaAs was caused by growth rate variations due to impurity concentration differences at dislocations.

Olsen and co-workers<sup>4,5</sup> found that relatively few dislocations were seen via x-ray topography in cases where the surface crosshatched pattern appears in optical viewgraphs. However, when the structures were severely mismatched ( $f > 2\%$ ), the layers usually did not exhibit the crosshatch morphology, but rather had an irregular surface. The loss of the regular surface morphology was considered the result of alloy inhomogeneities.<sup>5</sup> It was found that the crosshatch can be removed by a chemical-mechanical polish, but cannot be avoided in vapor-phase epitaxy (VPE) crystals unless lattice mismatch is kept below a critical value ( $f > 0.03\%$ ). This limited misfit range for the development of a smooth surface morphology is also observed in InGaAsP/InP heterostructures.<sup>6</sup> Olsen<sup>5</sup> suggested these crosshatched patterns are initially caused by accelerated crystal growth about misfit dislocations, and that once initiated, these "growth perturbations" will continue to propagate with growth as will any crystal surface defect.

Recently, surface crosshatched patterns have been reported<sup>7</sup> on annealed GaAs layers grown on Si substrates by metalorganic chemical vapor deposition (MOCVD). Nishioka *et al.*<sup>7</sup> suggest that the crosshatched pattern is a signature of dislocation motion during the thermal processing of GaAs/Si samples. They also found that crosshatched patterns were observed on relatively thick samples ( $> 2 \mu\text{m}$ ). This dependence on film thickness is correlated with the decreased threading dislocation density or etch pit density (EPD) in thicker samples. It is found that a clear,

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recognizable crosshatched pattern appears when the film has a relatively small etch pit density (EPDs) of  $\leq 2 \times 10^7 \text{ cm}^{-2}$ .

The wavelength and amplitude of the surface crosshatched profile was studied by Olsen.<sup>5</sup> In his thick samples, Olson found both the wavelength and amplitude of the crosshatch to be about  $1 \mu\text{m}$ . From the transmission electron micrographs of surface replicas, Matthews and Blakeslee<sup>8</sup> found that the amplitude of the surface crosshatched pattern in GaAsP/GaAs multilayers was about 30 nm or about 100th that reported by Olsen. They believed that this surface morphology was simply slip steps due to dislocation pileups. Surface crosshatch is also thought to be related to nucleation site of dislocation half-loops in InGaAs/GaAs strained layers grown by molecular-beam epitaxy (MBE).<sup>9</sup> Woodall *et al.*<sup>9</sup> suggested that the surface structure is the result of an enhanced growth rate at the dislocation half-loop nucleation sites, which are steps formed by the intersection of (111) slip planes with the (001) surface of the substrate.

An alternative explanation of the surface relief observed on compound semiconductor surfaces is associated with spinodal decomposition. Many ternary and quaternary III-V semiconductor systems exhibit spinodals.<sup>10-12</sup> Spinodal decomposition produces long-wavelength (100–300 nm) quasiperiodic composition fluctuations in  $\text{In}_x\text{Ga}_{1-x}\text{As}_y\text{P}_{1-y}$ , as has been observed by transmission electron microscopy.<sup>13</sup> It was suggested<sup>14</sup> that if a composition modulation has started to occur in a growing layer, the elastic deformation induced near the free surface should have important consequences on the subsequent growth of the layer.

The aim of this paper is to study the origin of the surface crosshatched morphology in strained compound semiconductors. The investigation is focused on strained InGaAs/GaAs growth using molecular-beam epitaxy (MBE). Nomarski interference microscopy, scanning tunneling microscopy (STM), and transmission electron microscopy (TEM) are applied to study the surface morphology, and both the threading and interfacial dislocations. The results suggest that the generation of the observed surface crosshatched morphology is directly correlated with the motion of interfacial misfit dislocations during strained III-V compound heteroepitaxial growth.

## II. EXPERIMENT

Molecular-beam epitaxial growth was performed in a three-chamber RIBER 2300 system. The strained InGaAs layers were grown on (001) undoped GaAs substrates. The substrates were initially solvent degreased. Mechanical damage resulting from polishing was removed by etching in a mixture of  $\text{H}_2\text{SO}_4\text{:H}_2\text{O:H}_2\text{O}_2$  (5:1:1). Surface oxides on the substrates were removed by a quick etch in concentrated HCl (1:1 with water). The substrates were then rinsed in de-ionized water and mounted on molybdenum sample holders with indium. Prior to growth, oxides were desorbed at  $600^\circ\text{C}$  under an  $\text{As}_4$  flux. A 0.25-mm GaAs buffer layer was first grown at  $600^\circ\text{C}$ , followed in

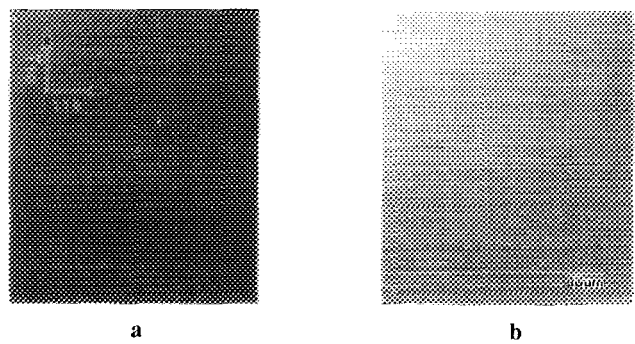


FIG. 1. Nomarski optical micrographs showing the surface morphology of strained  $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$  layers grown on GaAs at (a)  $400^\circ\text{C}$  and (b)  $520^\circ\text{C}$ .

$\text{In}_x\text{Ga}_{1-x}\text{As}$ , where  $x = 0.1-0.5$ , grown at a rate of  $1.0 \mu\text{m/h}$  at various thicknesses and substrate temperatures.

Surface morphology of epitaxial layers was studied by Nomarski interference optical microscopy. Specimens for cross-sectional TEM analysis were prepared by gluing together six  $5 \text{ mm} \times 5 \text{ mm}$  GaAs wafers face to face with epoxy, with the center two wafers containing the MBE-grown structures. The wafer blocks were sliced by a diamond saw along  $\langle 110 \rangle$  cleavage directions. The cross-sectional slices were ground, dimpled, and then thinned to electron-beam transparency by ion milling. Plan-view samples were dimpled from the substrate side before ion thinning. The specimens were examined in a JEOL 2000FX transmission electron microscope operating at 200 kV. Since the STM can only probe electrically conducting materials, the sample examined was a silicon-doped  $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$  layer grown on an *n*-type GaAs substrate.

## III. RESULTS

The effects of three experimentally accessible parameters on the surface crosshatched pattern have been studied: the growth temperature, the misfit strain, and the strained layer thickness. Figures 1(a) and (b) show the Nomarski micrographs of the surface morphology of  $1\text{-}\mu\text{m}$   $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$  grown on (001) GaAs substrates at  $400^\circ\text{C}$  and  $520^\circ\text{C}$ , respectively. A surface crosshatched pattern is clearly seen on the sample grown at  $520^\circ\text{C}$ , but the surface features are rather obscure on the one grown at  $400^\circ\text{C}$ . The heterointerfaces of both of these samples contain  $60^\circ$  misfit dislocations with similar dislocation spacings. However, since the nucleation of misfit dislocations is more difficult at low temperatures, the dislocation sources at high and low temperatures may differ.

Cross-sectional TEM micrographs of these two samples are shown in Fig. 2. The electron beam is along the [110] direction. Few threading dislocation were observed in the  $\text{In}_{0.25}\text{Ga}_{0.85}\text{As}$  layer grown at  $520^\circ\text{C}$ . In this case, almost all of the misfit dislocations were confined to the  $\text{In}_{0.15}\text{Ga}_{0.85}\text{As/GaAs}$  interface. It is believed<sup>15</sup> that the misfit strain can drive the tails of threading dislocations and dislocation half-loops to the edge of the epitaxial film.

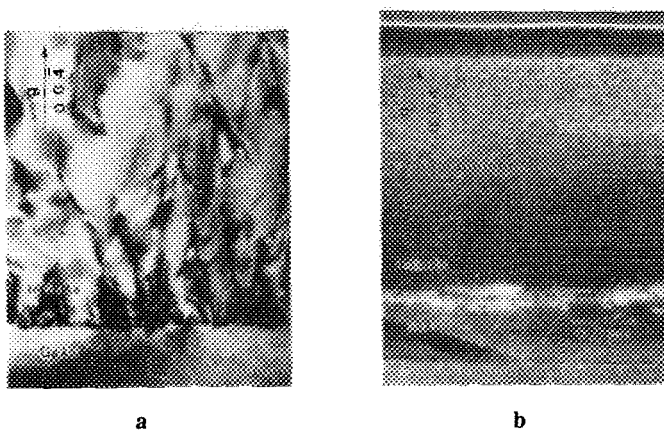


FIG. 2. Cross-sectional TEM micrographs of strained  $\text{In}_{0.25}\text{Ga}_{0.85}\text{As}$  layers grown on GaAs at (a) 400 °C and (b) 520 °C.

In contrast to the strained layer grown at 520 °C, those grown at 400 °C contain large numbers of threading dislocations. Most of the threading dislocations were 60° mixed dislocations which propagated along  $\langle 112 \rangle$  directions. This phenomenon is believed to be due to the insufficient thermal energy for the glide of the misfit dislocation tails. These dislocation lines (with inclined 60° Burgers vectors) stay in the glide planes and become “grown-in” threading dislocations. It is also very important to note that the strained layer which exhibits a clear crosshatched pattern has an overall smoother surface than the film which does not show this pattern (i.e., the one grown at 400 °C). A wavy surface profile (which may be too small to be observed in the wavelength range of optical microscope) can be clearly seen in the cross-sectional TEM micrograph shown in Fig. 2(a). This result is consistent with the results of a surface curvature study which used x-ray topography.<sup>3</sup>

Figures 3(a) and 3(b) show the surface morphology and a cross-sectional TEM micrograph of 1- $\mu\text{m}$   $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$  grown on GaAs at 520 °C. Instead of a surface crosshatched pattern like that seen in  $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$ , a rough textured surface is found on the  $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$

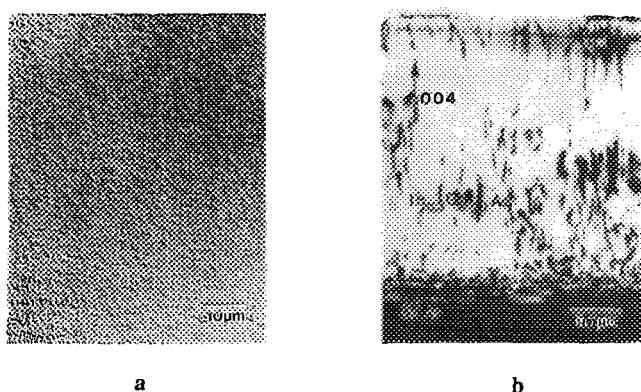


FIG. 3. (a) Nomarski optical and (b) cross-sectional TEM micrographs of strained  $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$  layers grown on GaAs at 520 °C.

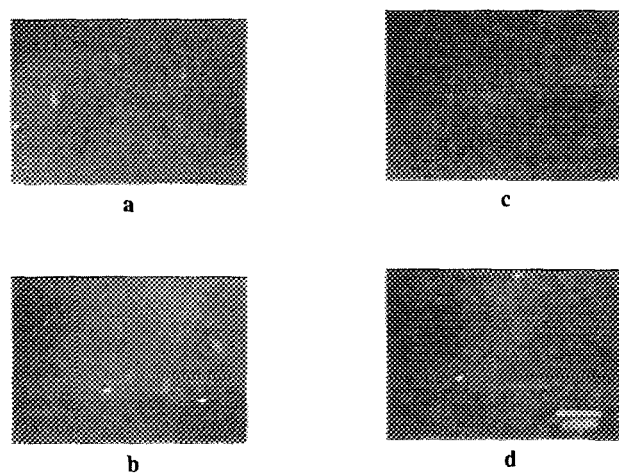


FIG. 4. Nomarski optical micrographs showing the surface morphology of the  $\text{In}_{0.14}\text{Ga}_{0.86}\text{As}/\text{GaAs}$  heterostructure with epilayer thicknesses of (a) 20 nm, (b) 40 nm, (c) 100 nm, and (d) 200 nm.

strained layer. In comparison to the  $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$  epitaxial layer shown in Fig. 2(b), numerous threading dislocations parallel to the growth direction were found in the  $\text{In}_{0.3}\text{Ga}_{0.7}\text{As}$  layer [Fig. 3(b)]. Since a 3D island growth mode is favorable in a lattice-mismatched system with a misfit larger than 2%,<sup>16</sup> the majority of the interfacial misfit dislocations are sessile-type pure edge dislocations formed through island coalescence.<sup>17</sup> These dislocations can thread up toward the film surface by a climb process. The majority of these threading dislocations are nearly perpendicular to the interface, instead of along  $\langle 112 \rangle$  directions. Moreover, the threading dislocations are not formed on  $\langle 111 \rangle$  glide planes and hence cannot glide up to the film surface to form slip steps.

Surface crosshatch was also studied as a function of the strained layer thickness. Figures 4 and 5 show the surface morphology and interfacial misfit dislocation network of  $\text{In}_{0.14}\text{Ga}_{0.86}\text{As}$  strained layers grown on a GaAs substrate

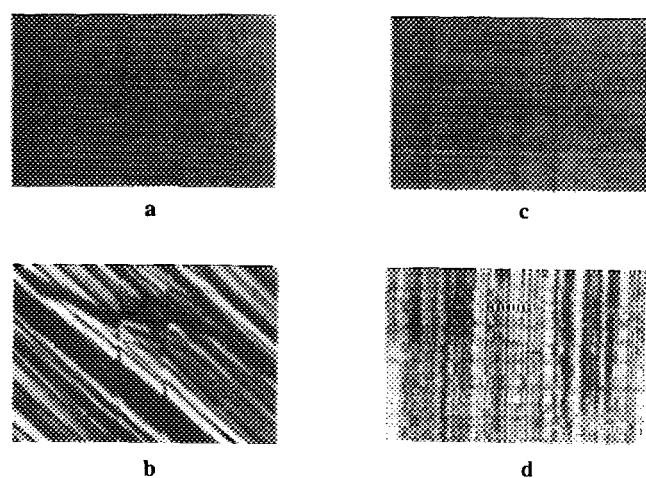


FIG. 5. Plan-view TEM micrographs showing the interfacial misfit dislocation network at the  $\text{In}_{0.14}\text{Ga}_{0.86}\text{As}/\text{GaAs}$  heterointerface with epilayer thicknesses of (a) 20 nm, (b) 40 nm, (c) 100 nm, and (d) 200 nm.

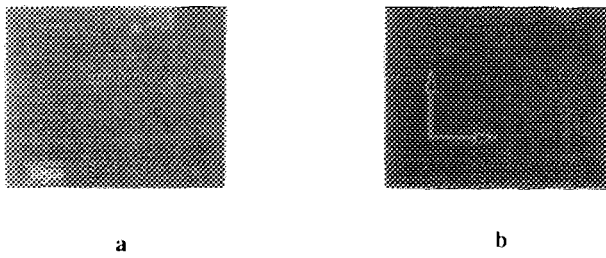


FIG. 6. Nomarski optical micrographs showing the surface morphology of strained  $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$  layers grown on GaAs at  $400^\circ\text{C}$  with layer thicknesses of (a) 100 nm and (b) 200 nm.

at  $520^\circ\text{C}$  with various thicknesses. The equilibrium critical layer thickness of this strained system as determined from the Matthews–Blakeslee analysis<sup>18</sup> is less than 10 nm. From the TEM results, it is found that the generation of the misfit dislocations starts at a layer thickness of 40 nm [Fig. 5(b)]. However, due to the small areas which are typically viewed in a TEM study, it is not possible to observe the very low dislocation densities which may be present during the early stages of misfit dislocation formation. At 100 nm thick, less than half of the misfit strain was accommodated by misfit dislocations. Most of the misfit dislocations are found to be mixed dislocations with Burgers vectors of the  $a/2\langle 110 \rangle$  type at  $60^\circ$  to the dislocation line. No surface crosshatched pattern was observed by Nomarski interference microscopy at this stage. A sharp surface crosshatched pattern appeared as the layer thickness was increased to 200 nm [Fig. 4(d)]. It is clear that the surface crosshatched pattern developed after the majority of the interfacial misfit dislocations were generated. The critical thicknesses below which the crosshatched pattern is not clearly observed was approximately  $0.2\ \mu\text{m}$  for this 1.0% mismatched growth. Similar experiments for the case of 0.5% mismatch illustrated that a clear crosshatched morphology developed at a layer thickness of about  $1\ \mu\text{m}$ . This result confirms that the presence of a surface crosshatch pattern is directly correlated with the presence of interfacial misfit dislocations.

As illustrated in Fig. 1, a poorly defined crosshatched morphology was found on the layers grown at relatively low temperatures. Figures 6(a) and 6(b) show, respectively, the surface morphology of 100- and 200-nm-thick  $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$  strained layers grown at  $400^\circ\text{C}$ . This figure shows that the surface crosshatch pattern becomes more pronounced as the strained layer thickness increases. When the growth temperature is lowered, greater layer thicknesses are required for the transition between metastable elastic-strained layers and misfit dislocation relaxed layers. As strained growth continues, large elastic stress fields are built up, enhancing the motion of dislocations and leading to the development of surface slip steps.

For the 100-nm  $\text{In}_{0.14}\text{Ga}_{0.86}\text{As}$  sample in Fig. 5(c), an *ex situ* annealing experiment was conducted at  $850^\circ\text{C}$  for 30 min under As overpressure. No surface crosshatched pattern was found on the sample after the annealing. Figure 7 shows that the majority of the misfit dislocations at

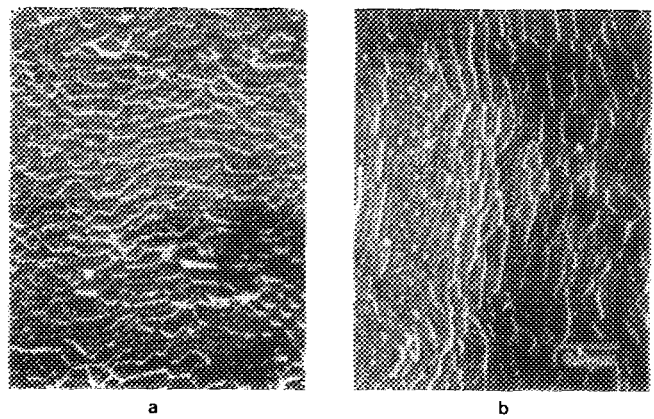


FIG. 7. Plan-view TEM micrographs showing the interfacial misfit dislocation network of the sample shown in Fig. 5(c) after annealing at  $800^\circ\text{C}$  for 30 min.

the heterointerface were edge-type dislocations. Although  $60^\circ$  mixed dislocations are more easily formed than pure edge dislocations at the strained interface, the large thermal energy available during annealing makes the formation of pure edge dislocations the favored mechanism to accommodate the rest of the elastic strain in the film. The formation of these pure edge dislocations may be either through the combination of two  $60^\circ$  mixed dislocations<sup>9</sup> or through a climb process. However, since no surface relief is formed during the annealing process, we can rule out dislocation formation through the glide process (which must be accompanied by the formation of surface steps).

Nomarski interference micrographs show that the wavelength of the surface crosshatch is about  $1\ \mu\text{m}$ , in agreement with Olsen's results.<sup>5</sup> The amplitude of the surface crosshatched profile was also studied by STM and cross-sectional TEM. Figure 8 shows the STM surface profile from which the height of the crosshatched morphology was found to be in the range 6–8 nm. Assuming that this profile is due to  $60^\circ$  mixed dislocations with 0.4-nm Burgers vectors, these results suggest that each surface step is due to the passage of 15–20 dislocations. This result is in reasonable agreement with the estimates of Matthews and Blakeslee.<sup>8</sup>

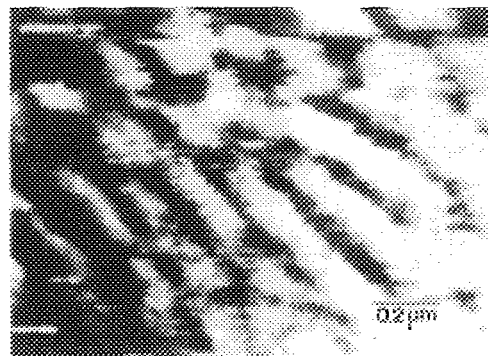


FIG. 8. Scanning tunneling micrograph showing the surface morphology of a  $0.3\text{-}\mu\text{m}$  Si-doped  $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$  layer grown on a  $n^+$ -GaAs substrate at  $520^\circ\text{C}$ . The amplitude of this surface steps are about 6–8 nm.



FIG. 9. Cross-sectional TEM micrograph of compositionally step-graded  $\text{In}_x\text{Ga}_{1-x}\text{As}$  layer grown on a GaAs substrate. Surface slip steps can be clearly seen at the top of the strained  $\text{In}_{0.5}\text{Ga}_{0.5}\text{As}$  epilayer.

From Fig. 2(b), the crosshatch profile cannot be observed by the cross-sectional TEM. The detail is too fine and is only visible in the optical microscope when the vertical contrast is enhanced by Nomarski interference. In order to observe clear crosshatched profiles in a cross-sectional TEM micrograph, a large number of dislocations have to glide to form large slip steps. It was also discussed earlier that no surface crosshatched pattern can be observed if the mismatch between the epilayer and the substrate is larger than 2%. Therefore, a step-graded  $\text{In}_{0.5}\text{Ga}_{0.5}\text{As}$  strained epilayer which had a 3.5% lattice mismatch was grown on a GaAs substrate. It can be seen in Fig. 9 that the majority of the misfit dislocations were confined in the strained interfaces. Since each step corresponded to only a 10% indium gradient ( $= 0.7\%$  misfit), the epitaxial growth was in the 2D layer-by-layer growth mode, and most of the misfit dislocations were  $60^\circ$  mixed dislocations. The specimen was tilted to bring the  $\{110\}$  zone axes perpendicular to the incident electron beam. The surface steps at  $\{111\}$  glide planes shown on the micrograph are along the  $\langle 112 \rangle$  direction. From the STM and cross-sectional TEM data, it is clear that the surface crosshatch pattern consists of slip lines arising from dislocations glide.

#### IV. DISCUSSION

When an epitaxial film is grown on a substrate with which it is misfit, dislocations form to relieve the misfit strain. Although this process first begins at the critical thickness  $h_c$  (which has been determined on the basis of dislocation theory), all of the strain is not immediately relieved as shown in film curvature studies. As the film continues to grow, its increased thickness results in increased strain energy, and hence additional dislocations form to relieve it. The intersection of these dislocations with the free surface causes slip steps, the height of which

is proportional to the number of dislocations which have cut the surface at each location.

All of the experimental data reported above may be interpreted in the framework of this simple picture. Since few dislocations exist in the film for  $h < h_c$ , no surface crosshatch is observed when the film is very small. For  $h < h_c$ , the dislocation density in the film begins to increase. Since the film contains a few dislocations at  $h \approx h_c$ , no surface crosshatch is observed. When the film thickness has increased to a sufficient level that the dislocation density within the film is large, surface crosshatch begins to appear. Further increases in film thickness result in the formation and motion of additional dislocations, thereby increasing the amplitude of the observed crosshatch pattern. Since the formation and motion of the dislocations in the film is a thermally activated process, increased driving forces must be applied in order to form the crosshatch pattern at lower growth temperatures. The additional driving forces may be found in the greater film thickness required to form the crosshatch pattern at lower growth temperatures.

Another source of the additional driving force required to move the dislocations which cause the crosshatch pattern at low growth temperatures is increased misfit strain. However, when the lattice mismatch of the strained epitaxial layer becomes larger than 2%, a rough, textured surface morphology is generally observed instead of the crosshatched morphology. This surface morphology transition may be traced to a change in the overall growth mode of the film. At large lattice mismatch strains, three-dimensional island growth is favored over the layer-by-layer growth mode. In the three-dimensional island growth mode, the interfacial misfit dislocations which form are of the pure edge type and are generated through island coalescence. These pure edge dislocations can thread up through a climb process and become grown-in dislocations during epitaxy. Since the Burgers vectors of these dislocations do not lie on  $\{111\}$  glide planes, they cannot glide to the  $(001)$  epilayer surface in order to leave the  $[110]$  and  $[\bar{1}\bar{1}0]$  slip steps on the surface which are characteristic of the crosshatched surface morphology. The surface crosshatched morphology is clearly observed in the strained epitaxy of films with misfit  $f < 2\%$  where two-dimensional layer-by-layer growth is the probable growth mode. In this case, the misfit strain is accommodated by the glide of  $60^\circ$  misfit dislocations tails through the elongation of substrate threading dislocations and/or dislocation half-loops.

The plot of epilayer thickness versus misfit strain shown in Fig. 10<sup>20</sup> summarizes the above results in a manner consistent with the interpretation just discussed. The shaded area represents the region in which the surface crosshatched morphology can be clearly observed. The surface crosshatch pattern forms for misfit strains smaller than approximately 2% and above a certain film thicknesses which are dependent on misfit strain. Two-dimensional layer-by-layer growth is presumed in this growth regime. As the lattice mismatch of the strained epitaxy becomes larger than 2%, a rough, textured surface morphology is generally observed instead of the crosshatched morphology.

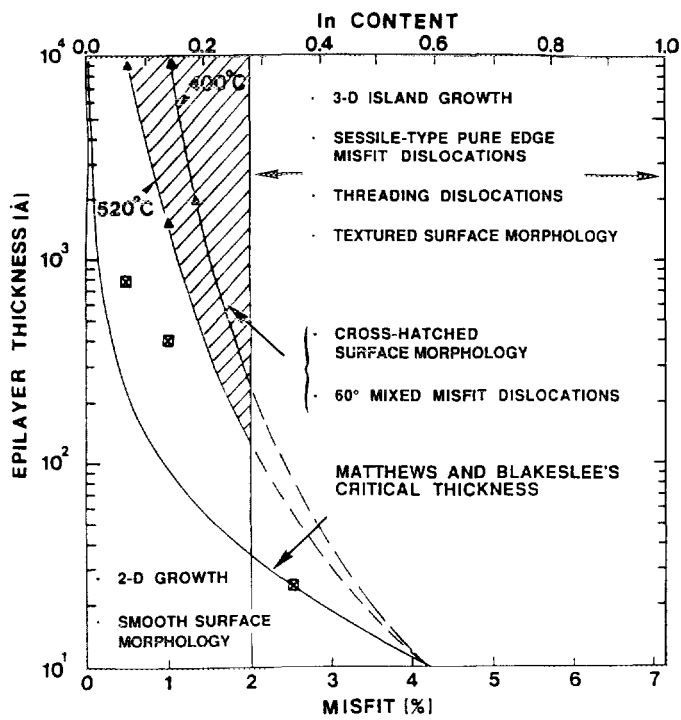


FIG. 10. A plot of epilayer thickness vs misfit in the InGaAs/GaAs heterostructure system. In the shaded area region of the diagram, crosshatched patterns are revealed.  $\square$  and  $\blacktriangle$  represent the layer thicknesses at which interfacial misfit dislocations and surface crosshatched patterns appeared, respectively.

Since the glide of dislocations in crystalline materials is a thermally activated motion,<sup>21</sup> the dislocation motion is further inhibited by the energy barrier as the growth temperature is lowered. Therefore, in comparison with the epilayers grown at 520 °C, the crosshatched morphology was observed at a much thicker epilayer thickness for the epitaxy at growth temperature of 400 °C, for which many glissile dislocations cannot overcome the local barrier of the frictional force during growth. It is unclear at this point whether the presence of a lower film thickness cutoff at  $h > h_c$  is attributable to limited resolution in the observation of film height variations or due to a lack of coherency of the dislocation motion when the dislocation density is very low.

Finally, we note that the observed surface crosshatch morphology is *not* due to spinodal decomposition in the present case. First, the phase diagram for the quaternary  $\text{In}_x\text{Ga}_{1-x}\text{As}_y\text{P}_{1-y}$  system at 630 °C presented by Smith and Burge<sup>11</sup> and Stringfellow<sup>12</sup> suggests that the tie-line of the miscibility gap is far from the InAs-GaAs pseudobinary system. Second, the quasiperiodic compositional contrast modulation of the InGaAsP alloy observed in (220) dark-field TEM images<sup>13</sup> lies in the [100] and [010] directions. This is a 45° rotation from the [110] and  $[\bar{1}\bar{1}0]$  surface crosshatched orientations observed throughout this investigation.

## V. CONCLUSIONS

The surface morphology of strained InGaAs/GaAs heterostructure has been studied. The crosshatched morphology on the surface of strained InGaAs epilayer grown on GaAs substrate occurs when the threading dislocation density in the epilayer is low. The morphology of the crosshatched patterns could not be observed when the lattice mismatch of the strained epitaxy was smaller than 2%, hence where 60° mixed misfit dislocations were generated. It is also clear that the surface crosshatched pattern developed after the majority of the interfacial misfit dislocations were generated. We conclude that the crosshatched pattern is constituted by surface slip steps of glissile dislocation motion in the strained epitaxial growth processing of the InGaAs/GaAs system.

## ACKNOWLEDGMENT

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