

# Characterization of unicompositional GaInP<sub>2</sub> ordering heterostructures grown by variation of V/III ratio

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Photoluminescence (PL) and photoluminescence excitation (PLE) spectroscopies are employed to investigate single heterostructures based on two GaInP<sub>2</sub> layers that have the same composition but different degrees of order on the cation sublattice. Four sample configurations are studied: two complementary single heterostructures, a more ordered layer grown on a less ordered layer and vice versa, and two single layers nominally equivalent to the constituent layers of the heterostructures. The degree of order of the two layers was controlled via the V/III ratio used during organometallic vapor phase epitaxial growth. From our measurements, the difference between the band gaps of the two layers is 20–30 meV. The PLE spectra show clearly that the emission comes from both layers of the heterostructures and that the PL is excited by direct absorption of the exciting light into each layer as well as the injection of carriers from the less ordered (higher band gap) layer into the more ordered (lower band gap) layer. The data clearly show that the heterostructures contain two layers, each very similar to the corresponding single layer sample. © 1997 American Institute of Physics. [S0021-8979(97)08322-9]

## I. INTRODUCTION

Atomic scale ordering of GaInP<sub>2</sub> grown by organometallic vapor phase epitaxy (OMVPE) in the CuPt structure is a well studied, if not well understood, phenomenon.<sup>1</sup> The most important effect of this ordering is a reduction in the band gap, theoretically predicted to be as much as 268 meV.<sup>2</sup> Ordering in this particular material is not only interesting from a scientific view, but may also have technological importance. This potential for applications stems from the fact that the degree of order, and hence the band gap, may be controlled by varying the growth parameters.<sup>3–5</sup> The idea of making heterostructures out of a material where the band gap changes while the atomic composition remains the same is an appealing one because it would remove the difficulty in lattice matching the layers (at both the growth and operating temperatures).

The two growth parameters that have been studied in terms of their potential for controlled variation of the degree of order are the growth temperature and the ratio of the partial pressures of the sources, the V/III ratio. Heterostructures<sup>6,7</sup> and quantum wells<sup>8</sup> based on a change in growth temperature have already been grown successfully. The use of variations in V/III ratio to grow heterostructures has a technical advantage over the use of growth temperature despite the fact that the degree of order can currently be controlled over a wider range with variation of growth temperature.<sup>9–11</sup> This advantage stems from the fact that changing the growth temperature requires an interruption of

the growth for a period of several minutes<sup>6,7</sup> to allow the temperature to stabilize. This interruption could have a negative impact on the quality of the interface in the heterostructure. In addition, the change in the growth temperature may result in a small change in the composition, partially negating the advantages of a heterostructure based on degree of order over a conventional heterostructure based on compositional changes.

## II. EXPERIMENT

The four samples investigated consisted of two single layers of ordered GaInP and two heterostructures, the latter made up of two layers grown to be identical to each of the single layers. The two heterostructures differ in the order in which the two layers were grown. A V/III ratio of 40 was used for the less ordered (higher band gap) layers and a V/III ratio of 160 was used for the more ordered (lower band gap) layers. All samples were grown by OMVPE on semi-insulating GaAs substrates misoriented 3° toward  $\langle 111 \rangle_B$ , using growth conditions intended to maximize the difference in degree of order due to a change in the V/III ratio, as described in detail elsewhere.<sup>12</sup> The epilayers are all composed of Ga<sub>x</sub>In<sub>1-x</sub>P, where  $x$ , as measured by x-ray diffraction, for all the layers was within 0.02 of its GaAs lattice matched value. These differences in composition are expected to produce energy shifts of less than 10 meV in the spectra. The growth temperature in all cases was 670 °C. Tertiarybutylphosphine (TBP) was used as the P precursor since it is expected to result in more abrupt interfaces than PH<sub>3</sub>.<sup>12</sup> The flow rates of the group III precursors, trimethylgallium (TMGa) and ethyldimethylindium (EDMIn), were

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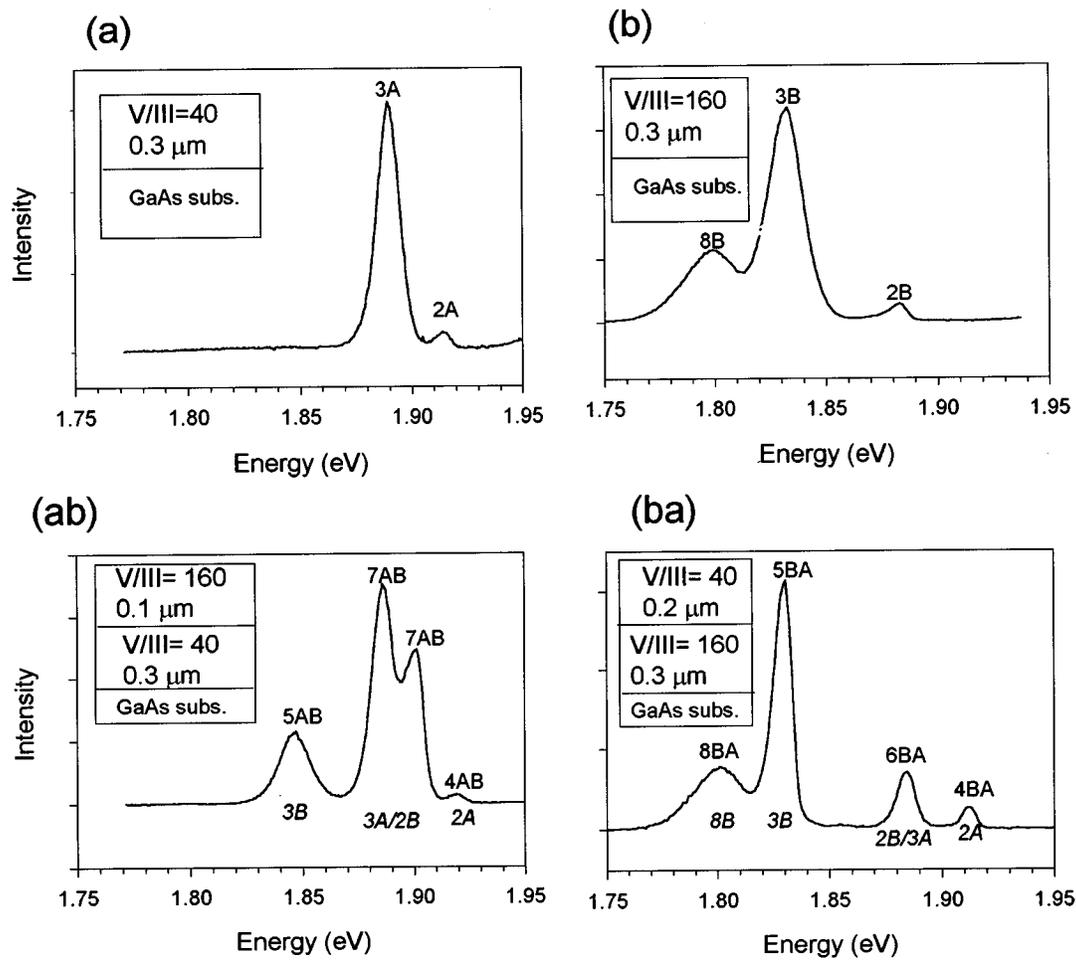


FIG. 1. PL spectra from the two homostructures (A,B) and two heterostructures (AB, BA) used in the study. A diagram of each of the structures, which shows the epilayer thicknesses, accompanies each plot. Labels correlate to subsequent PLE figure numbers and sample names. (a)  $V/III=40$ , less ordered; (b)  $V/III=160$ , more ordered; (ab) more ordered atop less ordered; (ba) less ordered atop more ordered.

held constant as the TBP flow rate was changed to produce the two heterostructures. Epilayers  $0.1\text{--}0.3\ \mu\text{m}$  thick were grown at about  $0.5\ \mu\text{m/h}$ . A diagram showing the thicknesses and order of growth accompanies the PL spectrum of each sample in Fig. 1.

Samples were mounted on a copper holder in a Janis cryostat and kept at 5 K in proximity to liquid helium. Photoluminescence (PL) was excited with  $5145\ \text{\AA}$  light from a Coherent Innova-70-4 argon ion laser or, for photoluminescence excitation (PLE), with  $6150\text{--}6800\ \text{\AA}$  light from a Coherent CR-599 dye laser with DCM (4-dicyano-methylene-2-methyl-6-p-dimethylaminostryryl-4M-pyran) dye. The exciting light was chopped with a mechanical chopper at the reference frequency of a Stanford Research Systems SR850 lock-in amplifier. The spectra shown in Figs. 1–6 were excited with an intensity of approximately  $0.5\ \text{W/cm}^2$ . The luminescence was dispersed with a  $0.85\ \text{m}$  Spex spectrometer and detected with a cooled GaAs photomultiplier.

### III. RESULTS AND DISCUSSION

#### A. Photoluminescence

PL spectra for all samples are shown in Figs. 1(a), 1(b), (ab), and (ba). Diagrams showing the  $V/III$  ratio and thick-

ness of each layer in the sample are included as insets. Throughout this document, the names A and B will refer to the less and more ordered single layers, respectively. The symbols AB and BA refer to the heterostructures where the letter order indicates the growth sequence. Lower case letters designate a subfigure containing data on the corresponding sample. The numerical labels above the peaks indicate corresponding figure numbers for the PLE spectra, excluding peaks 7 and 8 whose PLE spectra are not shown. The labels below the peaks in the spectra of the heterostructures indicate tentative identifications which can be made from a superposition of the two single-layer samples' spectra. More definitive assignments require examination of the PLE data. The high energy peaks in each of the single-layer samples, labeled 2A and 2B, are identified as due to excitons. This identification is made for two reasons: first, as will be discussed in the next section, they each have a PLE spectrum consistent with a strong coupling to a free-exciton absorption;<sup>13</sup> second they become more prominent in the PL spectrum at high excitation intensities as the lower-energy (and longer-lifetime) processes saturate.<sup>14</sup> The peaks to lower-energy are presumably associated with recombination through impurity levels. The exact origin of peak 8B, and

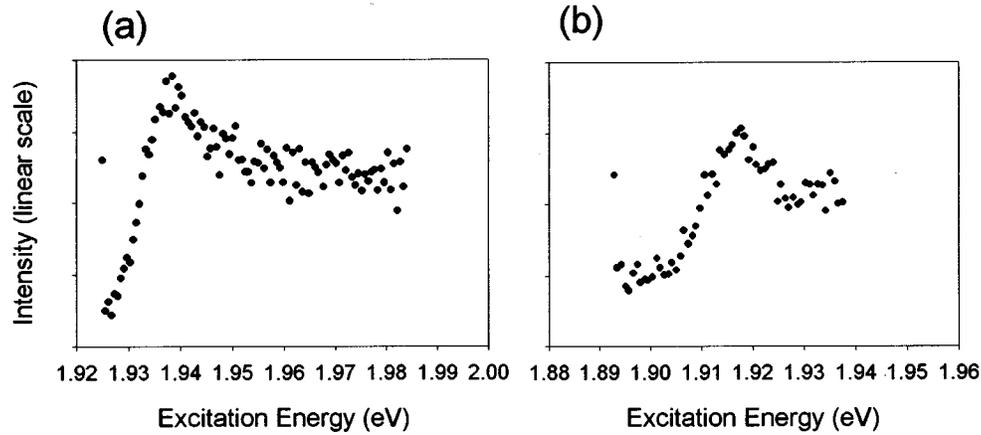


FIG. 2. PLE spectra detected on the highest energy (excitonic) emissions (2A, 2B) of the two homostructures. Note the peak associated with the free-excitonic absorption.

why it appears in the spectrum of the more ordered layer and not in that of the less ordered layer, is not known.

The PL spectra are complex. They contain multiple peaks whose relative amplitudes change drastically with excitation intensity. Also, some of the peak energies have a strong dependence on excitation intensity; this dependence is the so-called “moving emission” often seen in partially ordered GaInP<sub>2</sub> and previously attributed to spatially indirect recombination.<sup>15</sup> These factors make it difficult, in principle, to identify from which layer of the heterostructure the peaks originate, particularly since photoabsorption and carrier injection make impossible a knowledge of the effective excitation intensity in the heterostructures. Another potential problem is that some of the luminescence from the heterostructures could come from a process not occurring in either of the two single layer samples, presumably due to the interface between the two layers in the heterostructure. Despite these ambiguities, we will show that the combination of the PL spectra and the PLE spectra can be used to identify the source of each of the peaks in the PL spectra.

## B. Photoluminescence excitation

### 1. Single layers

The PLE spectra from the two single layers, detected on the peak of the excitonic emission in each layer, are shown in Figs. 2(a) and 2(b). The peaks in the excitation spectra near 1.94 and 1.92 eV, for samples A and B, respectively, are associated with excitonic absorption. Since the exciton binding energy is not expected to be significantly different for the two layers, this result indicates that the band gap difference between the two samples, as measured from the difference in the peaks of the exciton absorption energies, is 22 meV. Note that the presence of the exciton also indicates that the samples are relatively homogeneous on length scales greater than that of the radius of the exciton, approximately 70 Å.<sup>14</sup>

The PLE spectra from the next highest energy emission peaks in each of the two single layer samples are shown in Figs. 3(a) and 3(b). Of note in both spectra is the fact that there are two “steps” separated by 20–30 meV. The origin of these steps is not understood. However, the two steps will

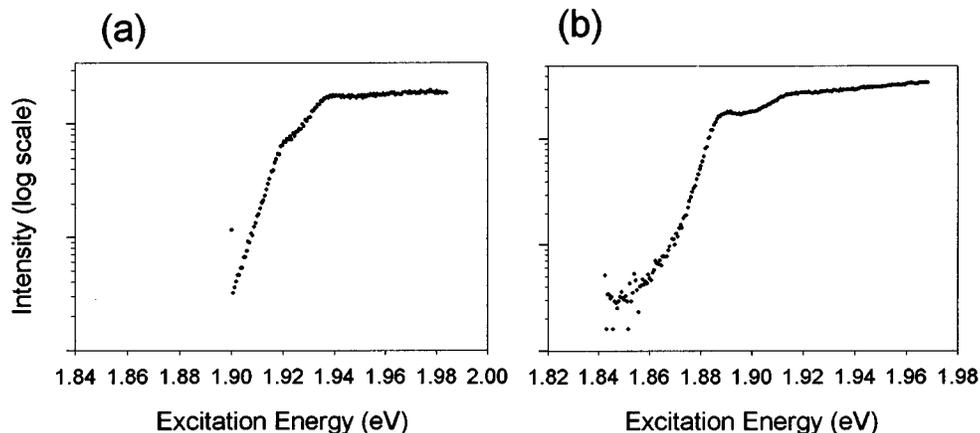


FIG. 3. PLE spectra for the two lower energy emission peaks (3A, 3B) of the homostructures: (a) peak 3A; (b) peak 3B. The spectra are used to identify emission peaks in the PL spectra of the heterostructures.

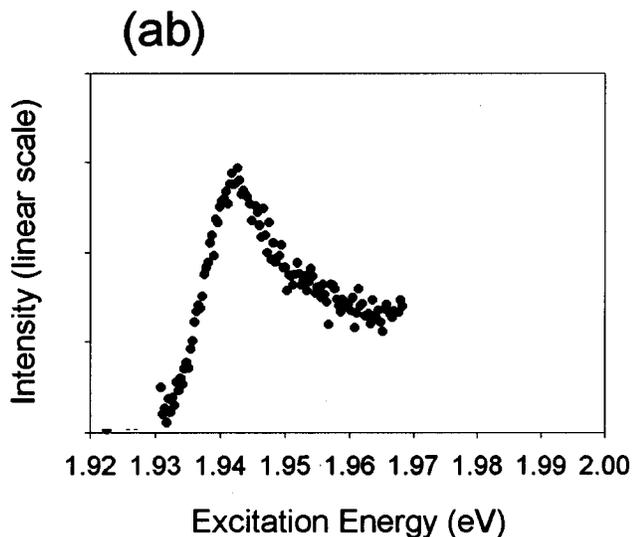


FIG. 4. PLE spectrum detected on the highest energy (excitonic) emission of sample AB (4AB). Note the similarity to the spectrum shown in Fig. 2(a). We conclude that the emission comes from the high band gap layer.

act as a “fingerprint” for emission from each of the nominally equivalent layers in the two heterostructures.

## 2. Heterostructures

In this section, the emission peaks in the two heterostructures will be identified in terms of the processes taking place in the two single layers. Complete quantitative agreement is not expected due to the fact that the emissions from each layer are dependent upon the power density of the exciting light, which is difficult to measure or control in the case of the heterostructures. In addition, many of the optical properties of partially ordered  $\text{GaInP}_2$  tend to be very sensitive to growth conditions, making complete quantitative reproducibility very difficult. In general, the tails of the PLE spectra from all the samples are broader than they would be for a homogeneous material at 5 K,<sup>13</sup> indicating some inhomogeneity in the samples.

The highest energy emissions from both heterostructures, labeled 4AB (1.919 eV) and 4BA (1.911 eV) on the PL spectra in Fig. 1, are unambiguously the excitonic emission from the less ordered (higher band gap) layer (1.913 eV from sample A). The PLE spectrum for sample AB, detected on this peak, is shown in Fig. 4. The PLE spectrum for sample BA, detected on peak 4BA, is very similar (not shown). Of note are the strong qualitative similarities to the PLE spectra taken from the single layer sample in Fig. 2(a). The two spectra are in quantitative agreement to within about 5 meV, peaking at 1.938 eV for the single layer and 1.942 eV for the heterostructure.

The peak labeled 5AB in Fig. 1(ab) can be identified from its energy (1.848 eV) as coming from the more ordered, top layer of sample AB. The emission is further identified as resulting from the same process as the peak in the corresponding layer labeled 3B (1.832 eV). Despite the fact that the discrepancy between the energies is the largest in this study (approximately 10 meV larger than the shift in the

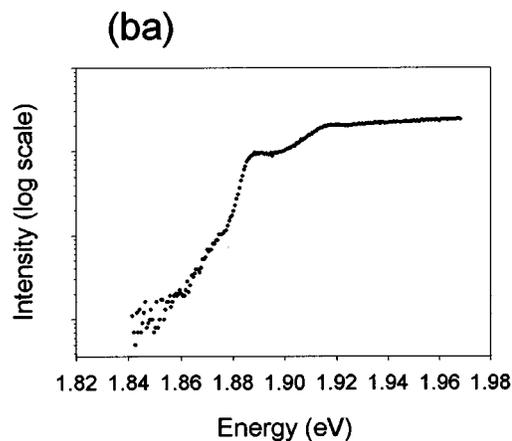
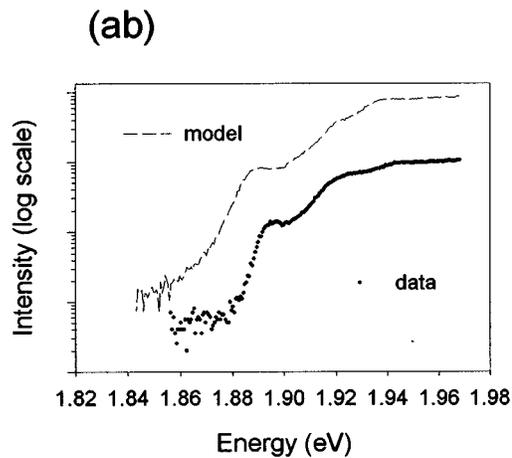


FIG. 5. PLE spectra for the emission peaks labeled 5AB and 5BA in Fig. 1: (ab) peak 5AB, (ba) peak 5BA. The emission is identified as coming from the smaller band gap layer. The dashed curve shown in Fig. 5(ab) is a weighted sum of two spectra from the homostructure samples. A multiplicative offset is used to separate the two curves.

excitonic emission and excitation spectra), analysis of the PLE spectra clearly supports this assignment and there are no other candidates in the PL spectra of the two single layers. Because peak 3B dominates the spectrum of sample B at low

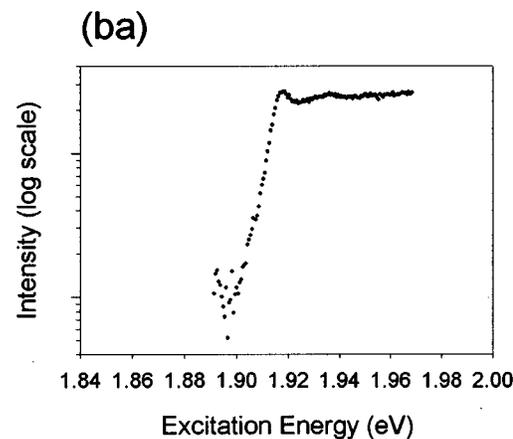


FIG. 6. PLE spectrum detected on the emission peak labeled 6BA in Fig. 1. The emission comes from excitons in the smaller band gap layer. The two peaks separated by  $\sim 15$  meV are due to the split heavy-hole and light-hole valence band.

injection levels, we conclude that the excited carrier density in the top layer of sample AB is low compared to the density in the single layer sample B under similar optical excitation conditions. Such a low density of injected carriers could be a result of a larger influence of (nonradiative) recombination at the surface of this thin layer. It is expected that since the top layer of sample AB is thinner by a factor of 3 than the single layer sample B, surface recombination competes more effectively with bulk recombination in the former. The PLE spectrum detected on peak 5AB is represented by the solid circles in Fig. 5(ab). The spectrum is qualitatively very different than that shown in Fig. 3(b), in apparent contradiction to the assignment made above. However, we propose that this difference arises from an additional method of exciting the luminescence: minority carriers can be injected into the lower band gap (top) layer after being optically excited in the higher band gap (buried) layer by light that is transmitted through the relatively thin ( $0.1 \mu\text{m}$ ) top layer. The dashed line in Fig. 5(ab) is an attempt to model this behavior as a weighted sum of a PLE spectrum from the more ordered single layer, shown in Fig. 3(b), and a spectrum from the less ordered layer, shown in Fig. 3(a), with the relative weight in the sum being the sole fitting parameter. A sum of 10 times the spectrum shown in Fig. 3(a) and (one times) the spectrum shown in Fig. 3(b) results in good qualitative agreement between the model and the actual PLE spectrum detected on peak 5AB, with an arbitrary additive offset. The fact that the dominant mechanism for exciting carriers in the lower-band-gap layer is absorption in the higher-band-gap layer followed by injection into the lower-band-gap layer is not unusual, and this mechanism has been shown theoretically and experimentally to be dominant in quantum well structures.<sup>16</sup> Optical excitation of the more ordered layer by luminescence emitted from the less ordered layer is not expected to be an important mechanism because it could not account for an order of magnitude increase over that provided by the original laser excitation. At most this optical excitation from the luminescence of the less ordered layer would be comparable to the excitation by the green light if the luminescence efficiency of the less ordered layer were near 100%.

The observation of injection of carriers from the high band gap layer into the low band gap layer is not only a good sign for the potential usefulness of heterostructures based on ordering, it also gives insight into the microstructure of the layers. The fact that a large fraction of the excitation spectrum for peak 5AB is due to injection of carriers from the buried layer indicates that the 5 K mobility of the minority carriers in the bottom layer has to be fairly large, despite evidence for inhomogeneities in the band structure discussed in the previous section. Specifically, the 5 K minority carrier diffusion length must be at least on the order of the thickness of the buried layer ( $0.3 \mu\text{m}$ ) for a significant number of the minority carriers optically generated in the less ordered layer to reach the more ordered layer. Since these materials are  $n$  type, this could result from the inhomogeneity in the band structure being mostly in the conduction band. This conjecture is in agreement with theoretical predictions,<sup>17</sup> which indicate that the conduction bands contribute the most to the band offsets between regions of different degrees of order.

The peak labeled 5BA in Fig. 1(ba) ( $1.830 \text{ eV}$ ), whose PLE spectrum is shown in Fig. 5(ba), is assigned to the same process as peaks 5AB ( $1.847 \text{ eV}$ ) and 3B ( $1.832 \text{ eV}$ ). The PLE spectrum for peak 5BA, unlike that of peak 5AB, is qualitatively very similar to that of peak 3B. This means that in the heterostructure with the more ordered, lower band gap layer grown first (BA), most of the carriers participating in the luminescence process (peak 5BA) in the more ordered layer are optically excited in that same layer, leading to an excitation spectrum very similar to that of the single layer (sample B). The fact that more carriers are excited into this process by optical excitation rather than injection from the higher band gap layer may be due to the fact that the more ordered layer in sample BA is about three times thicker than that of sample AB and hence absorbs more light. Note that if the relative weights are reversed in the sum of the two PLE spectra plotted as a dashed line in Fig. 5(ab), the result would resemble the data plotted in Fig. 5(ba), i.e., the high energy features would be lost. In short, we see evidence for carrier injection across the interface in the sample whose structure makes that observation most likely: a thin, small band gap layer in contact with a thick, high band gap layer.

Before discussing the other emission peaks in the heterostructures it is appropriate to summarize the results to this point. The identification of the PL peaks 4AB, 4BA, 5AB, and 5BA, from their PLE spectra is a demonstration that the heterostructures in fact were grown with two layers having optical properties very similar to those of the two single layer samples, as intended. Thus, from the standpoint of epitaxial growth, it is clear that variations in V/III ratio can be used to make unicompositional heterostructures. In addition, injection of minority carriers across the interface, a desirable effect for device applications, has been observed in one of the heterostructures.

The PL peak labeled 6BA in Fig. 1 ( $1.884 \text{ eV}$ ), with PLE spectrum shown in Fig. 6, is most likely due to excitonic emission from the more ordered layer, i.e., it has the same origin as the PL peak labeled 2B ( $1.882 \text{ eV}$ ). The PLE spectrum has a distinct rise at  $1.92 \text{ eV}$ , presumably due to free-excitonic absorption. The weaker feature shown at  $1.935 \text{ eV}$ , not observed in Fig. 2(b) due to poorer signal to noise ratio, becomes stronger (relative to the peak at  $1.92 \text{ eV}$ ) as the polarization of the exciting light is changed. Previous studies indicate such a difference arises from the splitting of the valence band in the ordered material.<sup>18,19</sup> These two peaks are therefore attributed to the splitting of the valence band of the more ordered layer and not the influence of the less ordered layer. Using the theoretical value<sup>2</sup> of  $62 \text{ meV}$  for the valence band splitting in the perfectly ordered material, the degree of order may be deduced from the measured splitting of the valence band assuming a linear relation between the two. We estimate the degree of order in the more ordered layer to be 0.25, where a degree of order of zero corresponds to the random alloy and unity corresponds to the GaP/InP [111] superlattice.<sup>19</sup>

The peaks labeled 7AB in Fig. 1(ab) ( $1.884 \text{ eV}$  and  $1.900 \text{ eV}$ ) are attributed to the same emission process as peak 3A

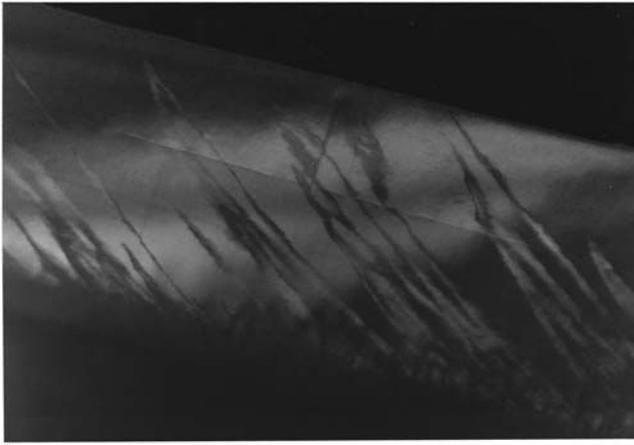


FIG. 7. TEM image of a heterostructure nominally identical to sample AB. Note the abrupt interface between the two layers.

(1.888 eV), i.e., they come from the less ordered layer. The PLE spectra (not shown) from each of the two peak positions labeled 7AB are very similar to the spectrum from the less ordered single layer shown in Fig. 3(a). The reason for the “splitting” of the peak in the heterostructure is not known.

The peak labeled 8BA in Fig. 1(ba) (1.801 eV) can be unambiguously identified as coming from the more ordered layer and corresponding to the emission labeled 8B (1.799 eV). This identification comes from the fact that the two peaks have very similar properties in terms of the dependence of their emission spectra on excitation intensity and the dependence of their excitation spectra on energy. The absence of this peak from sample AB can be explained by the thinness of the top layer of this sample. In this thin layer surface (nonradiative) recombination can compete more effectively with the radiative recombination taking place throughout the layer. This increase in the nonradiative recombination rate leads to a lower overall density of injected carriers in the top layer of sample AB than in the corresponding single layer sample, B. At lower excitation levels (not shown) the middle peak dominates the PL spectrum of sample B, and only this peak (peak 3B or peak 5AB) appears in the PL spectrum from the top layer of sample AB.

Figure 7 shows a transmission electron microscopy (TEM) image of a heterostructure with layers nominally identical to sample AB. Of note in the image is the abrupt interface between the two layers as is expected for these growth conditions.<sup>12</sup> The abruptness of the interface cannot be measured by our optical experiments, and has in the past been a difficulty in growing heterostructures based on ordering.<sup>12</sup> Also of note is the fact that the change in degree of order is difficult to see in the TEM image since neither layer is relatively close to being perfectly ordered or completely random. Examination of the transmission electron diffraction superspot intensities may be used to establish a difference in degree of order between samples grown with different V/III ratios.<sup>20</sup> However, the PL and PLE data are necessary in order to quantify the difference between the band gaps of the two layers of these heterostructures.

## IV. CONCLUSIONS

The PLE measurements performed on the series of samples studied here demonstrate the successful growth of two complementary single heterostructures based on a change in the V/III ratio during growth. The PLE spectra show that emission originates from both layers in each heterostructure. The luminescence from each layer can be excited by light absorbed in the layer that is emitting, or, in at least one case, by injection of (minority) carriers from the larger band gap layer into the smaller band gap layer. The luminescence properties of the two layers in each of the heterostructures are qualitatively similar to those of the single layers grown under similar conditions. With one exception, the luminescence and excitation peaks agree quantitatively to within 5 meV.

These conclusions demonstrate the usefulness of photoluminescence excitation spectroscopy in characterizing unicompositional heterostructure samples, and establish variation of the V/III ratio as a viable technique for heterostructure growth. In particular, in samples where more than one layer in the heterostructure emits light and the difference between the band gaps of the two layers is comparable to the full width at half maximum of the emission from the two layers, PLE can clarify PL spectra which are otherwise ambiguous.

## ACKNOWLEDGMENTS

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