Photoluminescence-linewidth-derived reduced exciton mass for $In_yGa_{1-y}As_{1-x}N_x$ alloys

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We report the measurement of the variation of the value of the linewidth of an excitonic transition at 4 K in $In_yGa_{1-y}As_{1-x}N_x$ alloys (1% and 2% nitrogen) as a function of hydrostatic pressure using photoluminescence spectroscopy. We find that the value of the excitonic linewidth increases as a function of pressure until about 100 kbar after which it tends to saturate. This change in the excitonic linewidth is used to derive the pressure variation of the exciton reduced mass using a theoretical formalism based on the premise that the broadening of the excitonic transition is caused primarily by compositional fluctuations in a completely disordered alloy. The variation of this derived mass is compared with the results from a nearly first-principles approach in which calculations based on the local-density approximation to the Kohn-Sham density-functional theory are corrected using a small amount of experimental input.

I. INTRODUCTION

Since the early work of Weyers *et al.*,[1] and Kondow *et al.*,[2] there has been considerable interest in the study of the growth, structural, electronic, and optical properties of $GaAs_{1-x}N_x$ and $In_yGa_{1-y}As_{1-x}N_x$ semiconductor alloy systems. These reports showed that small amounts of nitrogen, e.g., about 2% nitrogen in GaAs, could cause a band-gap energy reduction approaching 0.4 eV! This ability to tune the band gap energy between 1 and 1.4 eV (0.9 to 1.3 μm) makes it an attractive material system for optoelectronic devices.[2,3] The effect on laser thresholds by these unusual band-structure properties has also been discussed.[4] Furthermore, substituting a small amount of indium ($\sim 6\%$) for gallium in $GaAs_{0.98}N_{0.02}$ compensates for the strain effect caused by the addition of nitrogen thereby allowing lattice matching with GaAs, providing a strain free environment. This system is an ideal candidate for 1.3 μm lasers and high-efficiency multijunction solar cells.[1–5]

Before new devices based on this material system can be completely realized, information about the band structure, band-gap energy, etc., is necessary. In order to make predictions based on first-principles band-structure calculations, a knowledge of the band-gap energy and its pressure derivative are generally the first two desired pieces of information. The band structure of 2% nitrogen in $In_yGa_{1-y}As_{1-x}N_x$ and its pressure dependence have been studied by photoluminescence (PL) techniques and the large band-gap reduction is attributed to strong Γ-L and Γ-X mixing, resulting in an increased energy separation at zone center between the Γ-L and Γ-X GaAs conduction bands.[6] Good agreement between experiment and calculations based on the local-density approximation (LDA) was found.[6]

Because of random alloy fluctuations and the resulting large local variations in the band-gap energy, the carrier mobility for these types of structures is low, in the range of 100 to 500 cm²/Vs. Hence the traditional method of measuring masses by cyclotron resonance is difficult if not impossible. However, exciton reduced masses μ for 2% nitrogen $In_yGa_{1-y}As_{1-x}N_x$ alloys were determined[7] using three other experimental techniques, (1) exciton diamagnetic shifts measured by magnetoluminescence spectroscopy, (2) room-temperature photomodulated reflectance spectroscopy, and (3) PL peak energy for lattice matched $In_yGa_{1-y}As_{1-x}N_x$/GaAs quantum well structures. The 4 K ambient-pressure reduced effective mass μ for the 2% nitrogen $In_yGa_{1-y}As_{1-x}N_x$ samples was found[7] to be in the range of $0.13\,m_0$ to $0.15m_0$, where m_0 is the free-electron mass. Henceforth, the free-electron mass m_0 will be implicitly assumed when expressing values for effective masses. The reduced mass $\mu \sim 0.13$ for 2% nitrogen in $In_yGa_{1-y}As_{1-x}N_x$ is about a factor of 2 to 3 times heavier than that of GaAs. Recently, Skierbiszewski *et al.*,[8] using far-infrared radiation, studied the plasma frequency of an electron gas in *n*-type $In_yGa_{1-y}As_{1-x}N_x$ alloys, and found that the conduction-band mass varied from 0.16 at 1% nitrogen to 0.4 at 3.3% nitrogen. Another mass measurement, reported by Chen and his co-workers,[9] used optically detected cyclotron resonance to directly measure the conduction-band mass. These authors found conduction-band masses of 0.12 and 0.19, respectively, for 1% and 2% nitrogen in $GaAs_{1-x}N_x$. Both kinds of experimental measurements are in good agreement with the 2% nitrogen mass data presented in Ref. 7. It should be noted here that for both $GaAs_{1-x}N_x$ and $In_yGa_{1-y}As_{1-x}N_x$ alloys, all of these mass measurements indicate that the mass increases with increasing nitrogen concentration. The pressure dependence of the exciton reduced mass μ for the 2% nitrogen samples was also found[7] to be much greater than observed for the binary $In_xGa_{1-x}As$ system.[10] This large pressure dependence for the mass was also attributed to the

Γ-L and Γ-X mixing and at high pressures, where the repulsion between the Γ-like and X-like bands is greatest, the effect of mixing the heavier X-band mass with the lighter Γ-band mass leads to heavier and pressure dependent conduction-band mass.[7]

In this paper we report measurements of the pressure dependence of the exciton reduced mass for 1% and 2% nitrogen samples in epilayers of lattice matched $In_yGa_{1-y}As_{1-x}N_x$/GaAs using a technique based on the full-width-at-half-maximum (σ) PL linewidth of an excitonic transition in $In_yGa_{1-y}As_{1-x}N_x$ alloys. We find that the excitonic linewidth σ increases as a function of pressure until about 100 kbar after which it tends to saturate. This behavior of σ is used to derive the variation of reduced mass of an exciton μ with pressure in this alloy system using a theoretical formalism of excitonic linewidth in semiconductor alloys. In this formalism it is assumed that at low temperatures the broadening of the excitonic transition in relatively good quality alloys is primarily caused by the compositional disorder experienced by the excitons.[11] The extent of the excitonic charge distribution that depends on its reduced mass plays an important role in determining the value of σ. As will be discussed below, by assuming that the only contribution to the PL linewidth is a result of alloy fluctuations, the ambient pressure PL linewidth σ yields exciton reduced masses consistent with previously reported mass values.[7–9] These PL linewidth derived masses are 0.1 and 0.15, respectively, for 1% and 2% nitrogen in $In_yGa_{1-y}As_{1-x}N_x$. The variation of μ with pressure thus obtained is discussed in light of the first-principles LDA band-structure calculation presented in Ref. 6. It is also found that the pressure dependence of the σ-derived masses are in excellent agreement with that obtained from magnetoluminescence measurements.[7]

II. EXPERIMENTAL DETAILS

The $In_yGa_{1-y}As_{1-x}N_x$ structures were grown in a vertical flow, high-speed rotating disk, EM-CORE GS/3200 metalorganic chemical vapor deposition reactor using trimethylindium, trimethylgallium, 100% arsine and dimethylhydrazine as the gas sources. Dimethylhydrazine was used as the nitrogen source since it has a lower disassociation temperature than ammonia and has a vapor pressure of approximately 110 torr at 18° C. The unintentionally doped $In_yGa_{1-y}As_{1-x}N_x$ was nominally p type. $In_yGa_{1-y}As_{1-x}N_x$ films for Hall and optical measurements were grown on semi-insulating GaAs orientated 2° off (100) towards $\langle 110 \rangle$. The growth rate was 10 Å/s. The compositions for the samples reported here (nitrogen concentrations x~1% and 2%) were determined by calibration growths of $GaAs_{1-x}N_x$ and $In_yGa_{1-y}As$ along with double crystal x-ray diffraction lattice-constant measurements. The nitrogen composition was also measured by elastic recoil detection (ERD) measurements. By combining the results of the x-ray and ERD measurements, the nitrogen composition uncertainty for the 1% and 2% $In_yGa_{1-y}As_{1-x}N_x$ samples is estimated to be in the range of ± 0.15 to 0.2. For $In_yGa_{1-y}As_{1-x}N_x$, the lattice matching condition to the GaAs lattice constant for the indium and nitrogen concentrations y and x is $y \approx 3x$,[12] i.e., the approximate indium concentrations for 1% and 2% nitrogen

structures are, respectively, 3% and 6%. The lattice matched ($(\delta a/a) < 8 \times 10^{-4}$) films were grown at 600° C and 60 torr. A complete description of the details of the growth techniques and other important growth/annealing parameters has been discussed by Allerman *et al.*[12] A significant increase in PL intensity was observed from these films following a post-growth anneal. *Ex situ*, post-growth anneals were carried out in a rapid thermal anneal system under nitrogen using a sacrificial GaAs wafer in close proximity to the $In_yGa_{1-y}As_{1-x}N_x$ sample.[12] Transmission electron microscopy measurements indicate that the samples are random in composition and no evidence for clustering or phase separation was observed.[13]

The pressure was generated using a small BeCu piston-cylinder diamond-anvil cell, 8.75 mm in diameter and 12.5 mm in height.[14] The diamond-anvil cell was suspended from one of two concentric tubes. The internal tube acted as a ram so that the pressure could be modified by a drive mechanism at room temperature while the sample remained at 4 K. This probe also allowed us to use liquid helium as a pressure medium to assure a purely hydrostatic pressure. The preliminary 4 K 2% nitrogen $In_yGa_{1-y}As_{1-x}N_x$ pressure measurements reported in Refs. 6 and 7 were taken using methanol, ethanol, and water in a ratio of 16:3:1 for the pressure medium. For this report, we repeated the band-gap energy and PL linewidth measurements for the 2% nitrogen $In_yGa_{1-y}As_{1-x}N_x$ samples using the hydrostatic helium pressure cell and found no difference in their values from previous measurements and hence, in this paper, no distinction between these two separate measurements will be made.

The GaAs substrate upon which the sample was grown was mechanically lapped to a 1/2-μm-finish and thickness of 27 μm and then chemically etched with a 3% solution of bromine in ethanol to a final thickness of 23 μm. The shift in the fluorescence of a small chip of ruby placed in the pressurized volume was used to calibrate the pressure at 4 K with an accuracy of ± 0.5 kbar.[15] A single $600-\mu$m-diameter optical fiber, butted up against one of the diamonds, directed the nominally 1 mW power 514.5-nm-wavelength laser to the sample and also collected the PL signal from the sample. A beam splitter system was used to direct the PL signal to an optical monochromator. Depending on the band-gap energy, two liquid-nitrogen-cooled detectors were used to detect the PL signal. For the long-wavelength regimes, where the band-gap energies were near or below an energy of 1 eV, a NORTH-COAST EO-817L Ge-detector was employed, while for larger band-gap energies, a standard charge coupled device (CCD) array was used.

Typical ambient pressure, low-temperature (T = 4 K) PL spectra for nominally 1% nitrogen (right trace) and 2% nitrogen (left trace) in $In_yGa_{1-y}As_{1-x}N_x$ lattice matched to GaAs are shown in Fig. 1. The 4 K band gap energies are nearly 1150 and 1225 meV, which are significantly less than the 4 K GaAs band-gap energy $E_g \approx 1515$ meV. The PL linewidths $\sigma(x)$ are about 14 and 22 meV, respectively, for the nominally 1% and 2% nitrogen samples.

III. RESULTS AND DISCUSSION

Before we discuss how the variation of the exciton reduced mass with pressure is determined from the study of the
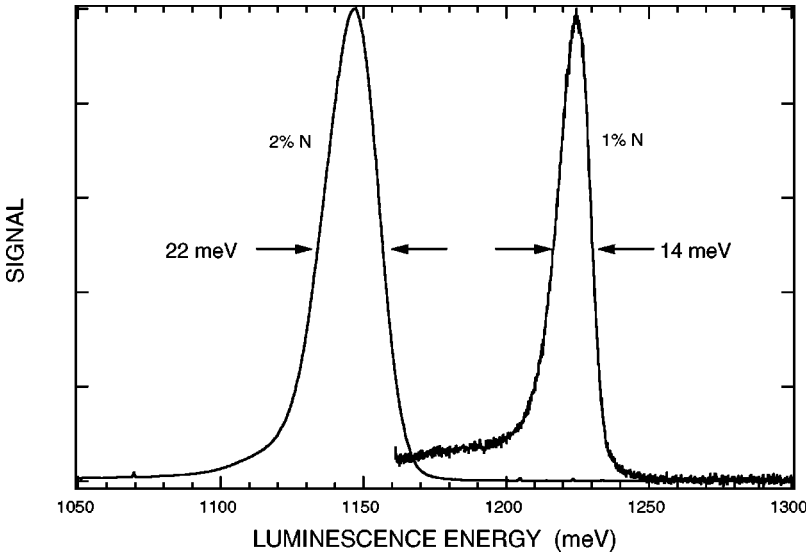
FIG. 1. Ambient pressure PL spectra at 4 K and for 1% nitrogen (right trace) and 2% nitrogen (left trace) in $In_yGa_{1-y}As_{1-x}N_x$. The linewidths are $\sigma(1\% \text{ nitrogen}) \approx 14$ meV and $\sigma(2\% \text{ nitrogen}) \approx 22$ meV.

behavior of σ with pressure, we briefly outline the basic ideas underlying the theory of excitonic linewidth in alloys. Excitonic transitions in semiconductor alloys, as observed in optical measurements such as PL, PL excitation, and modulated reflectance, are considerably broader than those observed in their binary components. The broadening mechanism has been attributed to compositional disorder, which is inevitably present in these kinds of systems. In high-quality alloys, this disorder is expected to be completely random. The physical origin of the excitonic line broadening due to compositional disorder lies in the fact that the average alloy composition inside the volume occupied by the exciton is different from that inside the volume of another exciton in a different spatial region of the alloy. Even though the global or average value of the alloy composition is fixed, excitons in different spatial regions in the alloy experience different local average values of the alloy composition. Within the framework of the virtual-crystal approximation (VCA), the conduction and valence-band edges sampled by the exciton are determined by the local alloy composition. Therefore, excitons in different spatial regions have different optical transition energies, thus leading to inhomogeneous broadening of the transition energy. Here we implicitly assume that the exciton localization is entirely a result of the band-gap energy variations due to the random alloy fluctuations. Because of the large values of the variation of band-gap energy with composition (see below), motional averaging and other linewidth contributions by the thermal motion of the free exciton, as discussed in detail by Lyo,[16] are neglected. Furthermore, because the valence-band offset between $GaAs_{1-x}N_x$ and GaAs for the 1% and 2% nitrogen alloys is small[6] the contributions to the alloy fluctuation theory of line broadening by fluctuating valence-band hole energies[16] is also ignored.

Based on this concept, several groups (see Lee and Bajaj,[11] and references therein) have calculated the variation of the excitonic PL linewidth $\sigma(x)$ as a function of alloy composition. They essentially calculate the probability of an exciton with volume V_{exc} experiencing an alloy composition x_{exc} about the mean value of x and relate it to the exciton transition energy using the VCA. In this model, the exciton PL line shape function f(E, x) is Gaussian, i.e.,

$$f(E,x)=[2\pi\sigma_0(x)]^{-1/2}\exp\left[-\frac{1}{2}\left(\frac{E-E_g(x)}{\sigma_0(x)}\right)^2\right], \quad (1)$$

where $E_g(x)$ is the average band-gap energy for the alloy with composition x. From the alloy fluctuation model for PL linewidths, σ_0 can be expressed as[11]

$$\sigma_0(x)^2=K^2 \ x(1-x)\left(\frac{V_0}{V_{exc}}\right)\left(\frac{\partial E_g}{\partial x}\right)^2. \quad (2)$$

In this expression V_0 is the smallest volume over which the composition fluctuation can occur and for the zinc-blende structure is equal to $(a_0^3/4)$, a_0 being the lattice constant; V_{exc} the volume of the exciton and in this paper is set to $(4\pi/3)r_{exc}^3$, where the exciton Bohr radius is $r_{exc}=(\hbar^2\kappa/\mu e^2)$, κ is the dielectric constant, e is the electronic charge, and μ is the reduced mass of the exciton. The term $\partial E_g/\partial x$ is the variation of band-gap energy E_g with alloy composition x. Depending on the exact definition of the exciton volume, the value of K in Eq. (2) varies between 0.32 and 1. For the analysis of the linewidth data reported here, we use K=0.4. This value of K has been derived by Zimmerman[17] and independently by Lee and Bajaj[11] using somewhat similar quantum-mechanical approaches that do not involve explicitly an ill-defined concept of the so-called exciton volume. In terms of $\sigma_0(x)$ and the Gaussian line shape f(E, x), Eq. (1), the full-width-at-half-maximum linewidth $\sigma(x)$ is

$$\sigma(x)=2\sqrt{2\ln 2}\,\sigma_0(x). \quad (3)$$

Thus from Eqs. (2) and (3), depending on the requirement, the exciton linewidth σ can be described in terms of the reduced mass μ or conversely, the reduced exciton mass μ can be described in terms of σ, i.e., $\sigma\propto\mu^{3/2}$ or $\mu\propto\sigma^{2/3}$. Using GaAs values for the dielectric constant $\kappa = 12.5$ and for the lattice constant $a_0=5.6\times10^{-8}$ cm, an expression for μ can be written in terms of $\sigma(x)$, x, and $\partial E_g/\partial x$ as

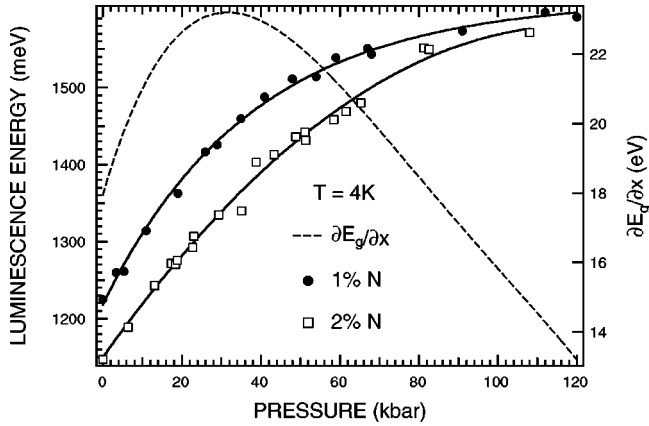$$\mu=\left(\frac{5.3 \ \sigma(x)}{\sqrt{x(1-x)}\dfrac{\partial E_g}{\partial x}}\right)^{2/3}. \quad (4)$$

FIG. 2. The pressure dependencies of the band-gap energy for the 1% nitrogen (filled circles) and 2% nitrogen (open squares) $In_yGa_{1-y}As_{1-x}N_x$ samples for T = 4 K. The smooth curves drawn through the data are an aid to the eye. The right-hand axis shows the pressure variation of $\partial E_g/\partial x$.

FIG. 3. The pressure dependence of σ for the 1% nitrogen (filled circles) and 2% nitrogen (open squares) at T = 4 K. The smooth curves drawn through the data are an aid to the eye.

Traditionally, values of the linewidths $\sigma(x)$ are calculated in terms of the exciton reduced mass μ and then compared with the experimental data (see for example Lee and Bajaj,[11] and references therein.) As mentioned above, the differences between the various theoretical treatments concerned the definition of the exciton volume. In this paper, we accept the validity of the alloy fluctuation model for the linewidth [Eqs. (2) and (3)] and use the experimental linewidth to infer the reduced exciton mass. From the PL spectra shown in Fig. 1 and with $K=0.4$ and $\partial E_g/\partial x \approx 20$ eV,[7] the exciton reduced mass μ for the 1% sample can be calculated from Eq. (4) with the result $\mu \sim 0.10$ for the 1% nitrogen structure, and for the 2% nitrogen sample, $\mu \sim 0.13$. These mass values ~ 0.13 for the 2% nitrogen in $In_yGa_{1-y}As_{1-x}N_x$ agree with the aforementioned experimental results[7–9] obtained by the more conventional optical determinations. This agreement between the calculated mass from the linewidth data and other mass determinations is thus the basis of this paper for using the pressure dependence of $\sigma(x)$ to obtain pressure dependent masses.

The 4 K pressure dependencies of the band-gap energy for the nominally 1% nitrogen (filled circles) and 2% nitrogen (open squares) $In_yGa_{1-y}As_{1-x}N_x$ samples are shown in Fig. 2. Some of the data for the 2% nitrogen structure are taken from Refs. 6 and 7 and some were taken with the helium pressure cell. The smooth curves drawn through the data are meant to be an aid to the eye. As can be seen from Fig 2, the separation between the 1% and 2% nitrogen alloys band-gap energies is less at high pressures than at ambient pressure. Similar results have also been reported by Shan, et al.[18] who measured the pressure dependence of the photomodulated reflectivity signal for several nitrogen concentrations in $In_yGa_{1-y}As_{1-x}N_x$ and $GaAs_{1-x}N_x$ alloys. Because of the repulsion between the Γ-bands and X-bands at high pressures, the difference between band-gap energies for differing nitrogen concentrations tend to diminish.[7,18] Thus if the alloy fluctuation theory for the pressure dependence of the linewidth σ is going to be used in order to obtain mass data, the pressure dependence of $\partial E_g/\partial x$ must be considered. The pressure dependence of $\partial E_g/\partial x$ can be extracted from the data shown in Fig. 2 or from the data reported in Ref. 18.
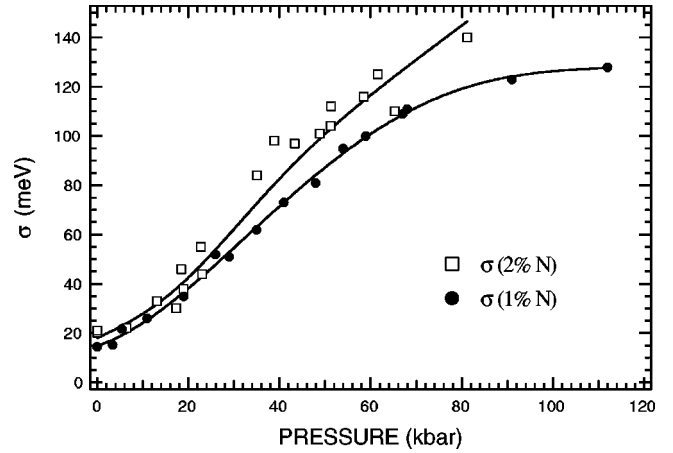
The right-hand axis of Fig. 2 shows the experimentally determined pressure dependence for $\partial E_g/\partial x$ from the data reported by Shan et al.[18] The experimental curve for $\partial E_g/\partial x$ is obviously sensitive to the accuracy of the knowledge of the nitrogen concentration in $In_yGa_{1-y}As_{1-x}N_x$ alloys. This kind of data for established and extensively studied alloy systems such as $Al_xGa_{1-x}As$ have been well documented in the literature.[19] However, for $In_yGa_{1-y}As_{1-x}N_x$, the *bowing parameter* for $\partial E_g/\partial x$ and also its pressure dependence are still being determined.[20–22] One reason for this problem is that an accurate determination of alloy compositions for quaternary alloy systems such as $In_yGa_{1-y}As_{1-x}N_x$ at low indium and nitrogen concentrations y and x is difficult.

The pressure dependence of σ for both the 1% (filled circles) and 2% nitrogen (open squares) $In_yGa_{1-y}As_{1-x}N_x$ samples is shown in Fig. 3. The smooth curves, drawn through both sets of data, are an aid to the eye. As can be seen in the figure, σ increase dramatically with pressure changing from about an ambient pressure value of 20 meV to greater than 100 meV for 100 kbar.

Using the σ versus pressure data shown in Fig. 3, and the $\partial E_g/\partial x$ data shown in Fig. 2, we calculate the dependence for exciton reduced mass μ as a function of pressure and the results are shown in Fig. 4. The solid circles are for 1% nitrogen values and the open squares are for the 2% nitrogen values. The stars are from the LDA calculation discussed below. The smooth curve is drawn through the data for the sample with 1% nitrogen. We find that the values of the reduced exciton mass μ increase with pressure till about 100 kbar and then tend to saturate and also that the pressure dependence of the 1% and 2% nitrogen samples are similar. The pressure coefficients are nonlinear, especially when compared to those found in $In_xGa_{1-x}As$-type alloys.[10]

It should be pointed out that the near-band-edge emission we observe is most likely associated with excitons bound by the local alloy fluctuations in which case the excitonic wave function is only slightly perturbed by these fluctuations. Therefore the theory of excitonic broadening due to alloy fluctuations, which depends on free exciton wave functions, is still applicable. Also the values of the exciton reduced
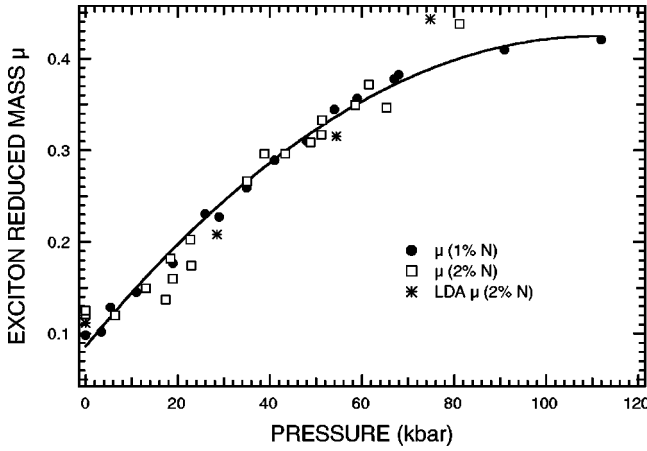
FIG. 4. Exciton reduced effective masses μ versus pressure for 1% nitrogen (filled circles) and 2% nitrogen (open squares) samples calculated from Eq. (4) using the linewidth data shown in Fig. 3 and the $\partial E_g / \partial x$ curve shown in Fig. 2. The smooth curve drawn through the 1% nitrogen mass data is meant to be an aid to the eye. The stars are the pressure dependent LDA calculated reduced mass.

mass we derive from excitonic linewidth represent upper limits as we have not taken into account other broadening mechanisms.

Because of our success in using first-principles electronic-structure calculations based on the local-density approximation to quantify the change in the band-gap energy with pressure,[6] we have performed LDA calculations for the pressure dependence of the conduction-band mass. Our results from these calculations are preliminary, and more complete calculations will be discussed, along with details of the method, in a future publication.[23] In summary, the Vienna *ab initio* simulation package (VASP)[24] was applied to the model of the $In_y Ga_{1-y} As_{1-x} N_x$ system detailed in Ref. 6, and the band masses were extracted using a carefully converged finite difference procedure. The well-known LDA band-gap error leads to an unphysically large repulsion between the conduction and light-hole bands at near-Γ k points. Since interaction of these bands is forbidden by symmetry at Γ, this results in calculated masses for these bands that are too light. The addition of nitrogen is believed to produce only minor changes in the GaAs valence bands, and therefore we can reasonably approximate the true $In_y Ga_{1-y} As_{1-x} N_x$ light-hole mass by the experimentally determined value for GaAs. Comparing this result with the light-hole mass obtained from our LDA calculations determines the numerical extent of the unphysical repulsion, which can then be used to correct the conduction-band mass. In order to compare with experiment, a reduced exciton mass is calculated by combining the corrected conduction-band mass with the heavy-hole mass (also approximated by its GaAs value). Using this procedure for a range of pressures requires knowing the pressure dependence of the GaAs valence-band masses, which are taken to be 1.0%/kbar and 0.5%/kbar for the light and heavy-holes respectively.[10] The reduced exciton masses found by applying this technique at four pressures are shown as stars in Fig.

4, and they show good agreement with the results derived from exciton linewidths. Given the extent of the approximations involved in the analysis of the LDA results, the quantitative agreement is better than would be expected and probably should not be taken too seriously. However, the qualitative agreement of these two independent methods provides a strong indication that both approaches are essentially valid.

Furthermore, we can qualitatively explain the pressure dependence of the conduction-band mass based on our LDA results. As discussed in Ref. 6, the band repulsion between the Γ-like and X-like bands at high pressure indicates that the amount of Γ-X mixing is increasing with pressure. This mixing, which would be symmetry forbidden in GaAs, is allowed in $In_y Ga_{1-y} As_{1-x} N_x$ due to the strong symmetry breaking induced by the nitrogen. Because the mass of the threefold degenerate X-point in GaAs is heavy ($m_{Xt} = 1.2, m_{Xl} = 0.27$), the conduction-band mass increases with pressure as the initially Γ-like conduction band mixes increasingly with the X-derived singlet. Similar conclusions have also been recently reported by Mattila *et al.*[25]

IV. CONCLUSIONS

We report an approach for measuring exciton reduced masses in semiconductor alloys. The reduced mass of the exciton is derived from the observed excitonic linewidth using a theoretical formalism, which is based on the premise that the broadening of the excitonic transition in alloys is caused only by the potential fluctuations. The $In_y Ga_{1-y} As_{1-x} N_x$ alloy system containing 1% and 2% nitrogen were studied using this technique. We find that the ambient pressure masses as well as the pressure dependencies are in good agreement with previously reported measurements. We observe that the value of the excitonic linewidth increases as a function of pressure until about 100 kbar and then tends to saturate. The variation of the reduced mass with pressure thus determined is examined in view of the results of a recent first-principles band-structure calculation using the local-density approximation where good agreement was found.

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