

# Photoreflectance investigation of hydrogenated (InGa)(AsN)/GaAs heterostructures

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**Abstract.** The optical response of as grown and hydrogenated  $\text{In}_{0.32}\text{Ga}_{0.68}\text{As}_{1-y}\text{N}_y/\text{GaAs}$  single quantum wells ( $y = 0, 0.027$ ) has been investigated from  $T = 80$  K to room temperature by photoreflectance. Three excitonic spectral features detected in the N free sample shift to lower energy in the N containing sample and back to higher energy upon H irradiation of the N containing sample. In the hydrogenated sample, a progressive change with increasing temperature of the nature of the lowest energy transition from an excitonic to a band-to-band character has been explained in terms of an increasing release of carriers from traps formed by H and N clusters. A reduction in the oscillator strength of the lowest energy transition and an increase in the binding energy of the heavy-hole exciton have been explained in terms of an increase in the electron effective mass upon N introduction into the  $\text{In}_x\text{Ga}_{1-x}\text{As}$  lattice.

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## 1 Introduction

Since their first proposal [1], InGaAsN/GaAs heterostructures have attracted much interest as GaAs-compatible high efficient emitters in the 1.3–1.55  $\mu\text{m}$  wavelength range. Nitrogen incorporation in (InGa)As leads to a large bowing of the band gap of the host lattice for increasing N concentration [2] and strongly modifies the hydrostatic pressure and temperature dependence of the band gap of the host material [3–16]. These effects have been accounted for in terms of a highly localized perturbation of the lattice potential due to the N atoms. In a perturbative approach [4], the N perturbation is reduced phenomenologically to the interaction between the level of an isolated N atom and the conduction band minimum (CBM). A more detailed theoretical model based on symmetry breaking considerations [17] predicts a sizable localized character of the CBM and the appearance of cluster states in the band gap. Recently, photoluminescence (PL) results [18,19] have shown that the optical properties of the (InGa)(AsN) system are strongly affected by hydrogen irradiation, which reduces the effective concentration of N atoms and reopens the material band gap.

In this paper, ground- and excited-state transitions were observed in as grown and hydrogenated (InGa)(AsN)

heterostructures by exploiting the phase-sensitive detection and the derivative-like nature of the photoreflectance (PR) technique. A detailed analysis of the PR line shape has then allowed to obtain information on the nature and the energy location of the critical points, to be compared with the predictions of recent calculations concerning the band structure of III-N-V alloys and to produce an experimental evidence of the increase of the exciton binding energy in the perturbed host lattice [20].

A blue-shift of all exciton related transitions is observed upon hydrogenation in N containing QW's. Therein, the binding energy of the heavy-hole exciton is found to increase largely. In the hydrogenated sample and for increasing temperature, the heavy-hole lineshape changes from that of a free exciton transition to that of a band-to-band transition. Meanwhile its spectral weight decreases with respect to that of the light hole transition. These effects are accounted for by the increase in the electron effective mass with N as well as by the increase in the free exciton screening when carriers are released from carrier traps formed in hydrogenated samples.

## 2 Experimental details

The heterostructures investigated have been grown on GaAs substrates by solid source molecular beam epitaxy.

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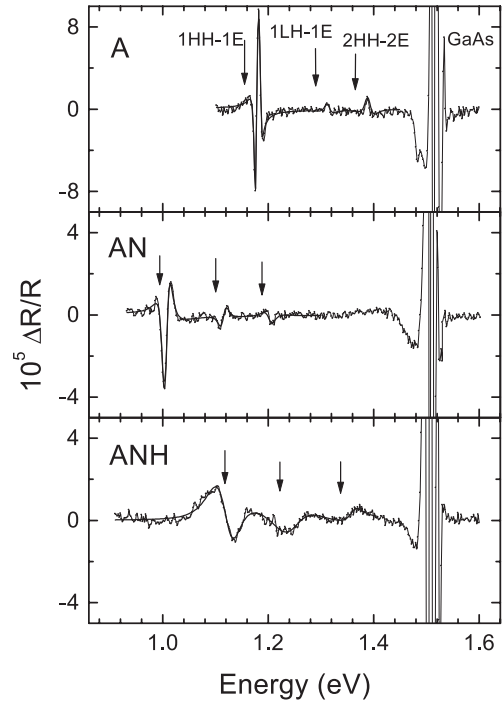
They consist of a 500 nm thick GaAs buffer, a 6 nm thick single  $\text{In}_x\text{Ga}_{1-x}\text{As}_{1-y}\text{N}_y$  well, and a 100 nm thick GaAs cap layer.  $\text{N}_2$  craking was obtained by using a radio frequency plasma source. The N concentration was determined by X-ray diffraction measurements. Post-growth treatment with atomic hydrogen was obtained by ion-beam irradiation from a Kaufman source. Further details about sample growth and H irradiation may be found elsewhere [10–12,18,19]. The as-grown and the hydrogenated structures ( $x = 0.32$ ;  $y = 0$ ,  $y = 0.027$ ) have been singled out from a set of previously well characterized samples [12,18,19]. The nitrogen concentration and H implanted dose ( $5 \times 10^{17}$  ions/cm<sup>2</sup>) were chosen with the intent of minimizing the degradation of PR signal and PL efficiency.

PR measurements were performed at near normal incidence in the 0.8–1.6 eV range, with spectral resolution of 1 meV. The standard experimental apparatus [21] operated with a 100 W halogen lamp as probe source. The excitation source was provided by a 2 mW He-Ne laser, mechanically chopped at a frequency of 220 Hz. The sample was mounted in thermal contact with the cold finger of a micro-miniature Joule-Thompson refrigerator that allows measurements in the 80–300 K temperature range.

### 3 Experimental results and discussion

Photoreflectance spectra taken at 90 K in the blank sample (A), the N-containing sample (AN), and the H-irradiated N containing sample (ANH) are shown in Figure 1. The signal associated to the GaAs band gap (buffer and cap layer) dominates the high-energy part of all sample spectra. In the low energy part of the spectra, three structures related to transitions in the InGaAsN quantum well are observed. The arrows in the figure mark the three transition energies obtained from a best fit of the PR spectra, as explained in the following. It is worth to point out here that i) the line shape models used in the fitting procedure are those of excitonic or band-to-band transitions in confined systems; ii) the first derivative of a Gaussian profile has been used for the dielectric function in order to take into account the inhomogeneous broadening [22] related to thickness and composition fluctuations of QW's.

Consistently with previously reported PL and PR results, [10–12] the first two structures are assigned to the 1HH-1E and 1LH-1E QW transitions and the third, highest energy structure is attributed to the 2HH-2E transition. The energies of the nHH-nE and 1LH-1E transitions reported in Table 1 at 90 and 300 K have been derived from a fit of the PR spectra that will be described in the following. At  $T = 90$  K, the energy separation  $\Delta_{1\text{H}2\text{H}}$  between the 1HH-1E and the 2HH-2E transition decreases from  $\Delta_{1\text{H}2\text{H}} = 212 \pm 5$  meV in sample A to  $198 \pm 5$  meV in sample AN, and returns to  $220 \pm 6$  meV in sample ANH, where H seems to counterbalance the effects of N on the material optical properties. At  $T = 90$  K, the energy separation  $\Delta_{1\text{H}1\text{L}}$  between 1HH-1E and the 1LH-1E transitions is equal to  $136 \pm 7$  meV in sample A and



**Fig. 1.** 90 K PR spectra of blank (A), N-containing (AN) and H-irradiated N-containing (ANH) samples. Arrows mark the QW-related transition energies as obtained by the analysis of the experimental line shape. PR spectra show evidence of the red-shift of all the QW PR features of the N containing sample (AN) with respect to the blank sample (A). In contrast, the same features display a large blue-shift in the hydrogenated N containing sample (ANH).

**Table 1.** Energy of the different excitonic (ex) and band-to-band (b-b) transitions as obtained by fitting equation (1) to the photoreflectance spectra at 300 K and 90 K. The experimental uncertainties on the energy of the 1HH-1E contributions are: 9 meV (2 meV) for the 300 K (90 K) excitonic contribution in sample ANH; 3 meV for the band-to-band contribution in sample ANH; 1 meV for all other cases at both temperatures. The experimental uncertainties on the energy of the 1LH-1E and 2HH-2E transitions are 3–5 meV for all other transitions [28].

Sample	T (K)	1HH-1E ex (eV)	1HH-1E b-b (eV)	1LH-1E (eV)	2HH-2E (eV)
A	300	1.108		1.237	1.319
	90	1.178		1.314	1.390
AH	300	1.106	1.113	1.230	1.308
	90	1.177	1.183	1.308	1.381
AN	300	0.946		1.046	1.153
	90	1.006		1.115	1.204
ANH	300	1.072	1.085	1.181	1.285
	90	1.126	1.141	1.246	1.361

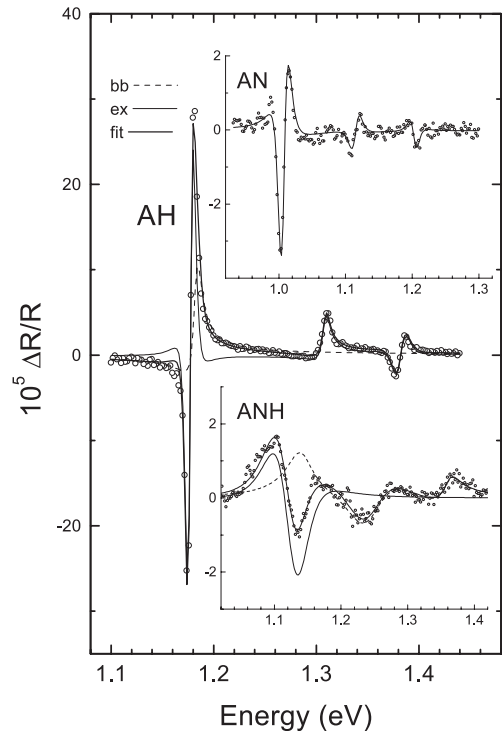
to  $109 \pm 4$  meV in sample AN. Recently, a modified 10-band  $\mathbf{k} \cdot \mathbf{p}$  Hamiltonian has been used to describe the band dispersion in a 7 nm thick  $\text{In}_{0.36}\text{Ga}_{0.64}\text{As}_{1-y}\text{N}_y/\text{GaAs}$  QW's, [23] namely, heterostructures quite similar to those investigated here. Therein,  $\Delta_{1\text{H}2\text{H}}$  is estimated to decrease from  $\sim 223$  to  $\sim 189$  meV and  $\Delta_{1\text{H}1\text{L}}$  to be equal to 120 meV for N concentration going from 0 to 2%, in very good agreement with present results.

In the N containing sample AN, the HH (LH) exciton transitions undergo at low  $T$  a red-shift of  $172 \pm 2$  meV ( $199 \pm 10$  meV). In the hydrogenated sample ANH, the HH (LH) exciton transitions blue-shift instead by  $119 \pm 3$  meV ( $128 \pm 8$  meV) with respect to the AN sample. This shift corresponds to a  $\sim 70\%$  (64%) recovery of the N induced reduction of the band gap of the N-free sample, in agreement with PL measurements [18,19]. A small variation of the PR structures transition energy is observed, instead, in the AH sample. As previously reported, these results as well as the increase of  $\Delta_{1\text{HH}2\text{H}}$  from sample AN to sample ANH can be accounted for by the formation of strong H-N bonds, which are favoured by the small size and high electronegativity of N [18,19,24]. This leads to a passivation of N and a reduction of its effective concentration.

An inspection of Figure 1 shows that the broadening parameter  $\Gamma$  of the strongest transition detected in the blank sample A increases roughly by a factor of two in sample AN (from 7 to 13 meV, from the fits at 90 K) and becomes about six times larger in sample ANH (43 meV). A similar behavior has been reported for the line width of the HH free exciton PL spectrum and accounted for by the increased disorder of the QW region of the samples produced by N introduction and further H-irradiation [19]. On the other hand, in the hydrogenated blank AH shown in Figure 2 the same broadening (7 meV) of the blank A sample has been found. Therefore, the simultaneous presence of N and H is needed to get the large broadening observed in the spectral features of sample ANH. Consistently with the inhomogeneous character of the broadening expected in QW transitions, a scarce dependence of  $\Gamma$  on temperature has been observed, except for sample ANH where the broadening as well as the 1HH-1E line shape (see below) exhibit an unusual behavior with increasing temperature.

In the case of sample AN the 1HH-1E transition essentially maintains the excitonic character it exhibits in N free InGaAs/GaAs QW's up to room temperature [22]. A similar behavior is found also in sample AH, where the H introduction increases the PR signal by a factor of 10 at 90 K and a factor of 3 at 300 K, thus permitting a more precise quantitative analysis of the signal. On the contrary, in the corresponding H irradiated sample ANH, the 1HH-1E structure by no means can be fitted by a simple excitonic transition, even at the lowest temperature. A band-to-band contribution, whose relative weight increases with  $T$ , should be added to the excitonic contribution in order to reproduce satisfactorily the experimental results. Moreover, the weight of the 1HH-1E transition decreases with respect to that of 1LH-1E transition upon N introduction in the lattice. The deconvolution of the best fit into band-to-band and exciton components is shown in Figure 2 for the 90 K spectra of samples AH, AN, and ANH. The experimental line shapes have been fitted in terms of the Aspnes formula [25]

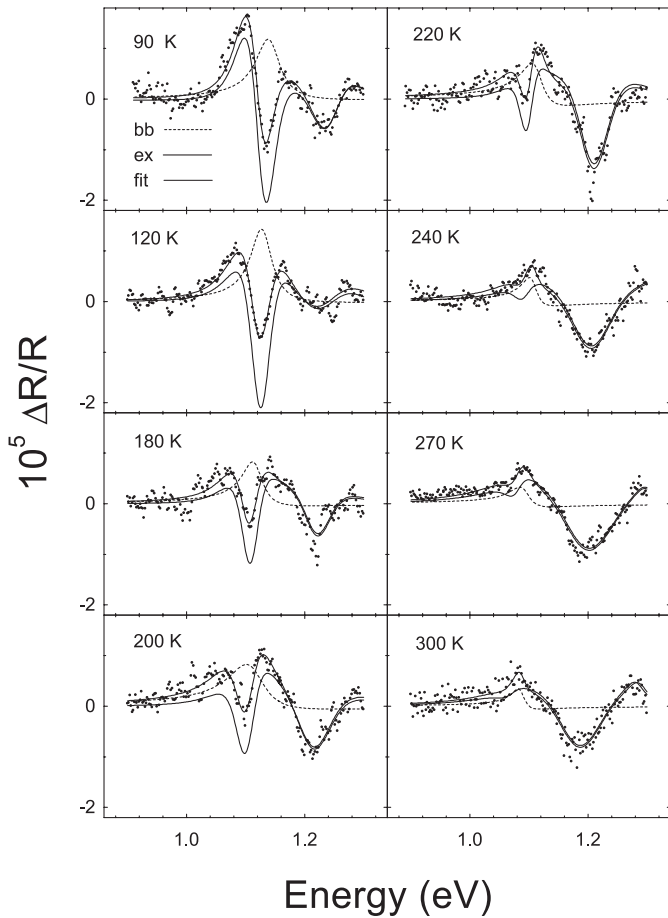
$$\Delta R/R = \text{Re}[\text{Be}^{i\varphi}(\hbar\nu - E + i\Gamma)^{-n}], \quad (1)$$



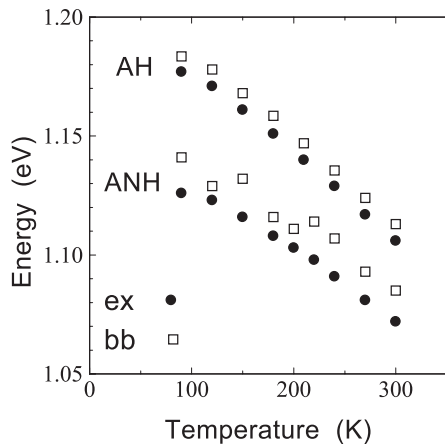
**Fig. 2.** Decomposition into exciton (continuous line) and band-to-band (dashed line) contribution of the 90 K QW structures of the samples AH, AN, and ANH. Solid lines indicate the best fit to the experiment (circles).

which has been used to reproduce electroreflectance and photorelectance spectra near a critical point in bulk semiconductors [25] as well as in confined systems [22,26,27].  $\hbar\nu$  is the photon energy of the probe beam and  $E$  is the critical point energy;  $B$  and  $\Gamma$  are the amplitude and the broadening parameter of the critical point, respectively;  $\varphi$  is the phase projection angle;  $n$  is an integer or half integer depending on the type of critical point. In the case of confined systems, exciton [22] and band-to-band [26] transitions have been satisfactorily fitted with values of the parameter  $n$  equal to 3 and 1, respectively. In the fits shown in Figure 2, two closely spaced structures with  $n = 3$  and  $n = 1$  have been used to reproduce the 1HH-1E signal in the AH and ANH samples. The other two QW signals are more noisy and in these as well as in the other samples can be equally fitted by using  $n = 1$  or 3 [28]. In the H free AN sample, the band-to-band contribution to the 1HH-1E transition is null within the experimental uncertainty. It is sizable, instead, in the same sample after hydrogenation and strongly increases with temperature, as shown in Figure 3 where the exciton component almost vanishes at room temperature.

The temperature dependence of the exciton (solid circles) and band-to-band (open squares) components of the 1HH-1E transition are shown in Figure 4 for the two hydrogenated samples, with and without nitrogen. The (mean) energy separation which characterizes the optical response of the two samples is quite different in the investigated temperature range. We point out that we can distinguish the two contributions to the PR signal of sample AH owing to the high signal-to-noise ratio of the



**Fig. 3.** Temperature evolution of the line shape of the 1HH-1E and 1LH-1E transitions for the hydrogenated N containing sample (ANH). Note the bleaching of the exciton component of the 1HH-1E signal as room temperature is approached. For sake of clarity the band-to-band component of the HH transition is separately reproduced with the dashed line, while its exciton component and the LH feature are both included in the continuous line.



**Fig. 4.** Temperature dependence of the exciton (solid circles) and band-to-band (open squares) component of the 1HH-1E transition for the hydrogenated samples.

signal after H-irradiation. The high relative intensity of the band-to-band component, instead, allows this distinction in sample ANH.

The experimental results for the ANH sample can be summarized as follows: i) the 1HH-1E transition has mainly an excitonic character at low  $T$ , which gradually transforms in a band-to-band character at room temperature; ii) the energy separation between these two contributions is an estimate of the exciton binding energy. It is equal to  $12 \pm 3$  meV from 90 K to 300 K. It should be noticed that this estimate is confirmed at intermediate temperatures, where the exciton and the band-to-band contributions to the signal have an equal weight; iii) the intensity of the 1HH-1E exciton transition significantly reduces for increasing temperature with respect to that of the LH transition.

In order to single out the effect of N, these results are compared with those found for the N free sample AH. Here, the PR signal was higher than in sample A or AN and the excitonic and band-to-band contribution to the 1HH-1E transition has been determined with high accuracy at all  $T$ 's. In sample AH, the exciton binding energy determined from the splitting of the two contributions to 1HH-1E is  $7 \pm 1$  meV, in excellent agreement with theoretical predictions [29,30]. In sample ANH this value increases to  $12 \pm 3$  meV. If one assumes that the dielectric constant does not change sizably upon the introduction of a small concentration ( $y = 0.027$ ) of N in the lattice, this change of the HH exciton binding energy is accounted for by a  $90\% \pm 40\%$  increase in the electron effective mass,  $m_e^*$ . An increase of  $m_e^*$  has been theoretically predicted on the ground of an increased localized character of the electron wavefunction upon N introduction in the InGaAs lattice [17]. The value found here agrees well with that (50%–100%) reported for similar QW structures on the basis of PL, excitation PL and photovoltage measurements [31,32].

Such an increase in the electron effective mass can also account for the reduction of the 1HH-1E transition intensity with respect to the 1LH-1E transition intensity. Indeed, in these heterostructures the LH effective mass is smaller than the HH effective mass [30,31]. Therefore, a change in  $m_e^*$  with N affects the heavy-hole exciton binding energy more than the light-hole exciton binding energy. In turns, an increase of  $m_e^*$  reduces the matrix element of the HH interband transition more than it does for the LH matrix element. Indeed, it has been shown that the  $\mathbf{k}\cdot\mathbf{p}$  thumb rule of a constant value of  $(1/m_E^* + 1/m_{HH}^*)^{-1}$  times the transition oscillator strength holds also in  $\text{In}_x\text{Ga}_{1-x}\text{As}_{1-y}\text{N}_y$  [23].

Let's finally account for the intriguing, strong change with temperature of the 1HH-1E transition. In AlGaSb/GaSb heterostructures, it has been shown that exciton can be partially screened by an increase in the impurity concentration in the well, thus favoring the reappearing of a band-to-band character in the QW transitions [33]. Moreover, both exciton and band-to-band transitions have been observed at room temperature on a doped GaAs/InGaAs/AlGaAs QW [34]. Therefore, the

change from excitonic to band-to-band transition reported in Figure 3 may be accounted for by the formation of trapping centers in the ANH sample due to the interaction of H with N clusters. These centers, indeed, supply more and more carriers as the temperature is increased and favor an exciton screening increasing with  $T$ . Their existence has been theoretically predicted and experimentally reported in (InGa)(AsN) [6, 17, 19, 35, 36], where H-N complexes have been invoked to account for a quenching of the PL signal in H irradiated heterostructures [18].

## 4 Conclusions

The effects of H irradiation on the optical properties of  $\text{In}_x\text{Ga}_{1-x}\text{As}_{1-y}\text{N}_y/\text{GaAs}$  single quantum wells have been investigated by monitoring the QW-related PR features. The observed blue-shift and temperature evolution of the PR spectra are in good agreement with PL results accounted for by hydrogen passivation of isolated N atoms and N clusters. The temperature evolution of the line shape of the observed transitions suggests a non radiative nature for the complexes H forms with N clusters. These results as well as the change of the HH exciton binding energy upon N introduction in the lattice are accounted for by an increase of the electron effective mass, in agreement with theoretical predictions and experimental results.

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