Direct-energy-gap dependence on Al concentration in $Al_x Ga_{1-x} As$

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The direct exciton energy at 2 K in $Al_x Ga_{1-x} As$ epitaxial layers (0.10 < x < 0.75) has been determined by optical transmission measurements. Microprobe analysis has been employed for the evaluation of the aluminum concentration. From these data, the direct-energy-gap dependence on x has been obtained; its comparison with the literature clearly shows that the relation $E_{gap}^{\Gamma}(x)$ commonly used in the optical determination of the alloy concentration must be revised. Contrary to older evaluations, our determination of the nonlinear contribution to $E_{gap}^{\Gamma}(x)$ agrees with the theoretically predicted values.

INTRODUCTION

Indirect procedures for the determination of aluminum composition x in the ternary alloy $Al_x Ga_{1-x} As$ requiring simple experimental apparatus and not overly time consuming are widely employed. These procedures exploit the dependence of x on easily measurable properties of the alloy, among which are the energy of the electronic states, the frequency of phonon modes and the lattice constant. The dependence of the energy of electronic states on x, determined through transmission or photoluminescence measurements, is the most commonly employed method of sample calibration because of its simplicity. Many dependencies on x of energies relative to electronic transitions are reported in the literature.¹⁻⁷ However, there are discrepancies among the published data and this is particularly evident in older determinations. Ten years ago the work by Dingle et al.³ seemed to have put an end to the problem. In fact, no other attempt to verify their results was made in the following years and the relation they found between E_{gap}^{Γ} and x has been widely used for calibrating $Al_xGa_{1-x}As$ samples since then. Very recently measurements performed on samples of $Al_x Ga_{1-x} As$ with low aluminum concentration (x < 0.5) (Refs. 4–7) put the work by Dingle *et al.* into question, while they give new importance to older works, such as, e.g., that by Monemar $et al.^2$

We believe that discrepancies among values determined by different authors are due to the reliability of the different methods employed for establishing the Al concentration in the alloy. In the literature x has been determined using different techniques: microprobe analysis, 1,2,4,5 Auger-electron spectroscopy, 3 x-ray diffraction, 6 and nuclear-reaction techniques. ⁷ For all these methods a correct determination of the alloy composition is only possible with accurate calibration techniques; some methods require further complex elaboration of the data. Raman spectroscopy^{8,9} can also be employed for determining the Al mole fraction in an indirect way.

We here present a new determination of the dependence of the direct energy gap on the aluminum concentration x, obtained through transmission measurements and microprobe analysis. This latter technique has the advantage of being a nondestructive and local analysis. Furthermore, it is possible to take into account the phenomena that can affect the results and, with a good choice of analysis conditions, it is relatively easy to determine x with fairly good precision.¹⁰ Transmission measurements have been adopted as a spectroscopic means of investigation for energy states in the alloy. Even if they require a laborious sample preparation, interpretation of the data is in general straightforward and unambiguous.

EXPERIMENTAL METHODS

We performed transmission measurements at 2 K on 12 samples of $Al_xGa_{1-x}As$ grown by liquid-phase epitaxy (LPE) and having Al concentrations x between 0.10 and 0.75. Our samples consisted of a GaAs substrate upon which a buffer layer of GaAs and a layer of $Al_xGa_{1-x}As$ were grown. In order to obtain samples suitable for transmission measurements, the thickness of the $Al_xGa_{1-x}As$ layer was reduced to $\sim 2 \mu m$ using standard etching techniques. Furthermore, for transmission measurements, GaAs was etched away on a region having a diameter of ~0.5 mm using standard etching solutions.¹¹

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Al concentration was determined using microprobe analysis. For each sample the analysis was performed at two electron-accelerating voltages (15 and 20 kV), both on the same place where transmission spectra had been recorded and right near it, where the substrate had not been etched away. We analyzed $K\alpha$ lines for the three elements of the solid solution. All the samples had been treated in a 25 wt. % solution of NH₃ in water, so as to remove any oxide on the surfaces.

The raw data were corrected for atomic number (Z), absorption (A), and fluorescence (F), using the ZAF method for x-ray intensity correction.¹⁰ Since our samples do not have a homogeneous structure in depth, for the determination of x we had to correct the results taking into account the finite thicknesses of the epitaxial layers. The main problem we had to face was that of fluorescence from Ga and As in the substrate. For these two elements, the ZAF correction should be employed in its most general form. Since the parameters for the fluorescence correction are not precisely known, we just used the $K\alpha$ line of aluminum for determining x.¹² In our case we have Al in the upper epitaxial layer only and fluorescence from Al in this 2-µm-thick layer is negligible. As a matter of fact, counts for Al $K\alpha$ line were the same, within statistical fluctuations, both where the epitaxial layer had no GaAs underneath and where the substrate had not been etched away. Thus we could adopt the ZAF method in a reduced form, using only the atomic number Z and the absorption A factors in determining corrections. As calibration standards for the microprobe, we employed one sample of GaAs, 300 μ m thick, and three samples of AlAs. The latter had been grown by molecular-beam epitaxy (MBE) and the thicknesses of their epitaxial layers were 1.1, 2, and 2 μ m, upon which a cap layer of GaAs of 100, 230, and 500 Å, respectively, had been grown, so as to reduce any hygroscopic or oxidation effect. We avoided contact of the AlAs standards with air as much as possible. We corrected AlAs counts for the GaAs cap and for the thickness of the epitaxial layer; for the AlAs standard we averaged the counts of the three AlAs samples. For each sample we determined x from data measured at both 15 and 20 kV on the two previously mentioned positions. Finally, the different xvalues found for each sample were averaged and the results found are reported in Table I. The error in x has been calculated from the uncertainties of the counts of the standards and from the spread of the x values for each sample.

In order to find a further confirmation of the x values, for one of our samples we performed Raman measurements. For this purpose we used the frequency difference between LO AlAs and LO GaAs phonons in the alloy and employed the results found by Saint-Cricq *et al.*⁹ The Al concentration in this sample was found to be $x = 0.40\pm0.02$, in agreement with our microprobeanalysis determination ($x = 0.387\pm0.004$).

For transmission measurements we used a standard experimental apparatus with a tungsten-filament lamp as a broadband source. Samples were mounted strain free in a He-bath cryostat. As a detector at the exit of the monochromator we employed either a cooled GaAs cathode

TABLE I. Al concentration and excitonic peak energies (2 K) of our samples as determined by microprobe analysis and transmission measurements. Values for GaAs (Ref. 14) and AlAs (Ref. 15) have been added. Experimental errors are also given. The weights calculated from these errors have been used in the fitting procedure for $E_{gap}^{\Gamma}(x)$ [Eq. (1)].

Al concentation x (at. %) Δx		Exciton energy E_{exc}^{Γ} (eV)	$\Delta E_{\rm exc}^{\Gamma}$ (meV)	Relative weight	
13.9	0.2	1.729	1	22	
25.7	0.3	1.884	2	16	
31.7	0.3	1.963	1	19	
36.4	0.6	2.040	1	4	
38.7	0.4	2.074	1	7	
43.8	0.4	2.149	3	6	
44.8	1.1	2.164	3	1	
51.5	0.3	2.262	3	8	
57.0	0.4	2.362	4	6	
61.4	0.8	2.444	4	2	
69.5	0.5	2.486	7	2	
72.9	0.5	2.613	3	4	
0		1.5152	0.1		
100.0		3.13	10	3	

photomultiplier tube connected to a lock-in amplifier, or an intensified optical multichannel analyzer. Our resolution was between 1 and 2.5 Å when using the cooled photomultiplier and less than 1 Å when using the optical multichannel analyzer. With this second experimental configuration we could determine the energy of the Γ excitons even in samples with $x \ge 0.6$, since the signal-tonoise ratio could be enhanced by several factors. The energies of the excitonic peaks and the relative errors determined in each sample are reported in Table I.

RESULTS

We recorded several transmission spectra for each sample in the frequency region of the direct energy gap (Fig. 1). We averaged the energy value of the absorption-peak positions recorded on different places of the layer and estimated an error in the determination of their spectral position. Energy position of excitonic peaks recorded on each layer could change according to the place of analysis by up to 7-8 meV. Such an effect can be attributed either to strain or stress in the layer itself or to nonhomogeneity in Al composition. The effect of the former cannot be calculated because we do not know either the nature or magnitude of the stress in the layer. The latter cannot be verified as a displacement of 7-8 meV can be caused by a small variation in Al concentration, $\Delta x \sim 0.006$, which is of the order of the precision we have in evaluating x. For the excitonic peaks we observed, the width was usually between 4 and 8 meV (full width at half maximum) with a tendency to increase for higher Al concentration. The exciton broadening could be explained by the increase in the aluminum concentration through the depth of the LPE layers



FIG. 1. Internal transmission spectra at 2 K of some samples vs energy difference from the excitonic peak energy.

 $(\Delta x \sim 0.005 \text{ per } \mu \text{m})$.¹³ As a result, no structure due to excited states of the free exciton could be observed and no experimental evaluation of the excitonic Rydberg could be performed.

The energy of the excitonic peak versus x is reported in Fig. 2 together with the value of the energy of the excitonic peak for GaAs by Sell, ¹⁴ and the only known value in literature for the excitonic peak at 2 K in AlAs. ¹⁵ We fitted these values, with weights reported in Table I, using polynomial functions. A straight line of slope 1.48 eV fits our data very well, but it gives a value for the energy of the excitonic direct gap in AlAs (2.99 eV) too small compared to the value 3.13 eV given by Monemar. ¹⁵ In order to fit the AlAs excitonic gap energy within its experimental error, we need a cubic dependence of energy on x. However, three fitting parameters are too many for the number of data points we have, so we propose

$$E_{\text{exc}}^{\Gamma}(x) = 1.5152 + 1.36x + 0.22x^2 \text{ eV}$$
 (1)

The constant term has not been fitted and it has been taken from the literature.¹⁴ The errors on the linear and quadratic coefficients are, respectively, ± 0.03 and ± 0.06 eV.

If we want to compare our data with curves reported in the literature for the direct energy gap versus x, we have to add the free-exciton binding energy E_b^{Γ} to the energy of the peak. Since the exciton binding energy is rather small [4.2 \pm 0.2 meV for GaAs (Ref. 14) and about 11



FIG. 2. Excitonic peak energy at 2 K vs Al concentration. We also report literature data relative to GaAs (Ref. 14) and AlAs (Ref. 15). Errors are given in Table I. The line reported is the fit of data as given by Eq. (1).

meV for AlAs (Ref. 15)], we can determine the exciton binding energy $E_b^{\Gamma}(x)$ interpolating linearly between these two values:

$$E_b^{\Gamma}(x) = 0.0042 + 0.007x \text{ eV}$$
 (2)

The relation between the energy gap at 2 K and the Al concentration is then

$$E_{gap}^{\Gamma}(x) = E_{exc}^{\Gamma}(x) + E_{b}^{\Gamma}(x)$$

= 1.5194 + 1.36x + 0.22x² eV (3)

[rounding errors make the linear coefficient identical in Eqs. (1) and (3)].

We can also determine the dependence of the energy gap on x at room temperature. The expression usually employed for the energy-gap dependence on temperature is that of Varshni:¹⁶

$$E_{gap}(T) = E_{gap}(0) - \frac{\alpha T^2}{T + \beta} = E_{gap}(0) - \Delta E_{gap}(T) .$$
 (4)

Different values of the parameters α and β can be found in the literature.¹⁷⁻¹⁹ Plotting curves of E_{gap}^{Γ} versus *T* from different authors,¹⁷⁻¹⁹ we can verify that the discrepancy among them is within 15 meV. This is true not only for curves of E_{gap}^{Γ} versus *T* referring to GaAs, but also for AlAs: the curve given by Monemar¹⁵ for AlAs is in agreement (within 10 meV) with those given by Thurmond¹⁷ and Waters¹⁸ for GaAs.

In view of the smallness of these differences and of the errors we have on the linear and cubic coefficients in Eq. (3), we take a constant value for $\Delta E_{gap}^{\Gamma}(300 \text{ K}, x)$ equal to that of GaAs:¹⁷

$$\Delta E_{\rm gap}^{\Gamma}(300 \text{ K}, x) = 0.096 \text{ eV} .$$
 (5)

(We took for the AlAs direct gap the same variation in energy as for the free exciton, because Monemar gives the temperature dependence for the latter only. This way we assume the excitonic Rydberg to be the same at 0 and 300 K, neglecting effective-mass and dielectric-constant variation with temperature.)

After transferring our results [Eq. (3)] to 300 K, we obtain the dependence of direct energy gap on x:

$$E_{gap}^{\Gamma}(300 \text{ K}, x) = E_{gap}^{\Gamma}(0 \text{ K}, x) - \Delta E_{gap}^{\Gamma}(300 \text{ K}, x)$$
$$= 1.423 + 1.36x + 0.22x^{2} \text{ eV} . \quad (6)$$

DISCUSSION

We compare our results with those given in the literature. We shall only discuss the works in which the direct energy gap has been determined through optical measurements and the aluminum concentration has been directly measured. Further, we shall indicate in parentheses the experimental methods employed in each work.

We start considering the excitonic gap at 2 K [Eq. (1), Fig. 2]. In the literature, data are reported only for Al concentrations up to $x \sim 0.4-0.5$. Indeed, the Γ exciton has not been observed in transmission measurements so far on samples having higher values of x, and in photoluminescence measurements only structures relative to the X indirect gap can be observed for x > 0.5. We compare our results with data by Dingle et al.³ (transmission-Auger-electron spectroscopy), by Lambert et al.⁶ (photoluminescence—x-ray diffraction), and by Kuech et al.⁷ (photoluminescence-nuclear-reaction techniques and microprobe analysis). In the range of Al concentrations for which they report their results (x < 0.5), the energy of the excitonic peak depends linearly on x. The slope of the straight line given by Kuech et al. is 1.455 eV, and fits our data very well; the value given by Lambert et al. is smaller (1.34 eV). There is much stronger disagreement with Dingle et al.,³ from whose data we can estimate a slope of 1.20 eV. In particular, we observe that the discrepancy between this last result and ours is ~ 0.1 eV at $x \sim 0.45$. Such a discrepancy might be due to the particular method Dingle et al. used for calibrating x, as discussed recently by Kuech et al.⁷

For the direct energy gap at 2 K we compare our determination [Eq. (3)] with those by Monemar et al.² (transmission—microprobe analysis), Dingle et al.³ (transmission—Auger-electron spectroscopy), and Oelgart et al.⁵ (photoluminescence—microprobe analysis). All these determinations, together with ours, are reported in Fig. 3 or Table II. The first two works cover the whole range of Al concentrations while the last one is relative to



FIG. 3. Energy gap at 2 K vs Al concentration. Solid line has been obtained in this work [Eq. (3)]. Data relative to other authors are reported also: Monemar *et al.* (Ref. 2) (squares) and Dingle *et al.* (Ref. 3) (dashed line).

x values lower than 0.52. Our data compare favorably with those of Oelgart *et al.* (the slope of their straight line is 1.45 eV), as they do with those of Monemar *et al.* over the whole range of Al compositions, in spite of the greater errors on their values. The determination by Dingle *et al.*³ confirms the tendency they already showed for the excitonic gap. Their data are systematically below ours (with a discrepancy up to ~0.2 eV in energies or ~0.1 in x). It is interesting to note that other $E_{gap}^{\Gamma}(x)$ data which are difficult to interpret are reported by Arthur *et al.*²⁰

The room-temperature direct gap is of particular practical importance. Our evaluation [Eq. (6)] has to be compared with some of the already mentioned works (Monemar et al.² and Oelgart et al.⁵) and with two others, Berolo et al.¹ (electroreflectance—microprobe analysis) and Miller et al.⁴ (photoluminescence—microprobe analysis), together with the commonly adopted relation by Casey and Panish.²¹ We still confirm good agreement between our relation and the data by Oelgart et al. and Monemar et al., and also with those given by Miller et al. Data reported by Berolo et al. do not seem to agree so well. The discrepancy between our determination and that by Casey and Panish is similar to that we had at 2 K with the one by Dingle et al.³ All these data are reported in Fig. 4 and/or Table II.

TABLE II. Determination of the direct-energy-gap dependence on the Al concentration reported in the literature. The spectroscopic techniques, together with the methods for the determination of x, are indicated. Only the data for which the authors give an explicit numerical relation $E^{\Gamma}(x)$ are reported; a, b, and c are the coefficients of the polynomial $E^{\Gamma}(x) = a + bx + cx^2$.

Ref.	Experimental method	Т (К)	a (eV)	b (eV)	c (eV)	Determination of x	x range
Berolo ^a	electrorefl.	300			0.26	microprobe	0 < x < 1.0
Miller ^b	photolum.	300	1.42	1.45	-0.25	microprobe	0 < x < 0.45
Oelgart ^c	photolum.	300	1.425	1.35		microprobe	0.15 < x < 0.52
		2	1.514	1.45		•	
Lamberg ^d	photolum.	2		1.34		x-ray diff.	0 < x < 0.45
Kuech ^e	photolum.	2	1.512	1.455		nucl. react. and microprobe	0 < x < 0.5
Casey ^f	collect. of data	300	1.424	1.247			0 < x < 0.45
			1.656	0.215	1.147		0.45 < x < 1.0
This work	transmission	2	1.5194	1.36	0.22	microprobe	0 < <i>x</i> < 1.0

^aReference 1.

^bReference 4.

^cReference 5.

^dReference 6.

^eReference 7.

^fReference 21.



FIG. 4. Energy gap at 300 K vs Al concentration. Solid line is the result of our determination [Eq. (6)]. Other data reported are those by Berolo *et al.* (Ref. 1) (circles), Monemar *et al.* (Ref. 2) (squares), and Casey and Panish (Ref. 21) (dashed line).

In Table II we give a summary of the numerical values reported in the literature. All data relative to x determinations performed employing microprobe analysis are in reasonable agreement, and the coefficient relative to the linear term in x lies between 1.35 and 1.45 eV. The consistency among results of authors who employed microprobe analysis with different and independent calibration procedures confirms the validity of our choice and of our results. Raman measurements performed on one of our samples verifies our microprobe analysis.

Our determination confirms, among others, those by Oelgart *et al.*⁵ and Kuech *et al.*⁷ and, in particular, they suggest a value of x relative to the crossover between the Γ direct gap and the X indirect gap at 2 K lower than the commonly accepted one (~0.43 given by Dingle *et al.*³). Anyway, accurate measurements of the X indirect gap are needed.

As previously discussed, our data are best interpolated by a nonlinear polynomial in x. The small value we found for the quadratic coefficient c (0.22 ± 0.06 eV) agrees with what was predicted theoretically.²²⁻²⁴ Starting from both the dielectric model²² and pseudopotential calculations,^{23,24} the bowing parameter c is found to be very small. Compositional and positional disorder are small in absolute value and do not seem to contribute significantly to the bowing of the curve. Using the relation given by Hill²³ for c and data for covalent radii by Van Vechten *et al.*,²⁵ we find, for $Al_x Ga_{1-x} As$, c=0.045eV. Thus our experimental determination of c is not far from the theoretically calculated value and supports the hypothesis adopted in the theoretical determination, particularly the validity of the virtual-crystal approximation for this alloy.

CONCLUSIONS

Data on $E_{gap}^{\Gamma}(x)$ from the literature do not agree, par-ticularly those by Dingle *et al.*³ and Monemar *et al.*,² who, performing optical measurements of the same kind but employing different methods for calibrating x, get contradictory results. In order to find a more reliable determination, we made optical measurements on LPEgrown samples where the Al concentration was determined employing microprobe analysis. The good quality of samples gave the opportunity to determine Γ -exciton energies from transmission measurements at 2 K for alloy compositions up to x=0.73. In order to reduce to a minimum the error on the determination of x, we were particularly careful in the following two points: (i) we performed the microprobe analysis on the same place where transmission measurements had been done; (ii) the procedure for determining x took into account the particular structure of our epitaxial layers; we analyzed only the Al characteristic radiation in order to reduce ZAFcorrections.

Our results confirm those by Monemar *et al.*² and other recent results of photoluminescence measurements. As a consequence, the commonly used relation proposed by Casey and Panish²¹ and by Dingle *et al.*³ have to be revised, in particular for (i) greater slope of the curve E_{gap}^{Γ} versus x; (ii) lower value of Al concentration relative to Γ -gap-X-gap crossover; and (iii) very small or nonexistent bowing of $E_{gap}^{\Gamma}(x)$, in agreement with theory.

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tribution from the substrate. We found disagreement between this experimental difference and its value determined using standard corrections (Ref. 10). This point will be discussed in a forthcoming paper.

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