Codoping of wide gap epitaxial III-Nitride semiconductors

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Codoping in p-type, epitaxial GaN grown by metal-organic vapour phase epitaxy (MOVPE) was investigated. An enhancement of hole concentration was observed experimentally in p-type GaN:Mg codoped with oxygen donors. The hole concentration of GaN:Mg codoped with oxygen increased super-linearly from 8×10^{16} to 2×10^{18} cm⁻³ upon increasing the oxygen dopant partial pressure. A factor of 3–5 enhancement of hole concentration was measured for a fixed oxygen partial pressure during the growth of p-type GaN:Mg. However, when Si was codoped with GaN:Mg, the hole concentration remained constant. Current experimental results are compared with the existing theory of codoping in GaN.

Keywords: GaN, codoping, p-type conductivity.

1. Introduction

The attainment of high-conductivity wide gap compound semiconductors is central to their utilisation in optoelectronic devices. While n-type GaN with carrier concentrations as high as 8×10^{19} cm⁻³ has been achieved [1–4], hole concentrations are typically more than an order of magnitude lower [1,5-7]. Factors limiting p-type conductivity in III-V nitrides include: limited solubility of acceptors, high ionisation energy of acceptors, and compensation by impurities and native donor defects [8]. Indeed, it has been proposed that there is a natural doping limit for these wide gap materials [1,9]. This limit arises from the formation of compensating native defects. The formation energy of donor defects in p-type material is lowered upon decreasing the Fermi level. For a certain Fermi level, the energy required to form the defect is offset by the energy to occupy it with electrons. This pinning Fermi level is approximately 4.9 eV below the vacuum level in III-V semiconductors as determined experimentally [1]. Using first principles total energy calculations, the equilibrium pinning energy has been calculated and it is in excellent agreement with experiment [9]. Equilibrium calculations indicate highly doped GaN would be semi-insulating.

As to the identity of these compensating defects in p-type GaN, they include the nitrogen vacancies V_N , the associated vacancy complexes such as V_N -Mg_{Ga} [5,10] and V_N -H [11] and impurities such as H, Si, and O [8]. The question of course arises: can GaN be doped higher than the equilibrium doping limit? One approach that has been proposed is codoping with both acceptor and donor impurities

[12–15]. Codoping can lead to enhanced solubility by forming defect complexes with large formation energy. Furthermore, through complex formation and screening effects there is the potential for lowering the ionisation energy.

There have been a number of experimental studies of codoping in GaN and its alloys. Some of the existing experimental reports are listed in Table 1. A few important conclusions can be drawn from the data presented in Table 1. First, bulk crystals grown at high temperature are semi-insulating as was shown for GaN crystals unintentionally codoped with both Mg and O [16]. The presence of Mg-O pairs was detected by UV reflectance spectroscopy. Semi-insulating behaviour is expected based on the doping limit rule, since the pinning level is deep within the gap for GaN.

Codoping of GaN thin films with Be and O [17] by molecular beam epitaxy (MBE) and with Mg and O by MOVPE [6,18] and MBE [19] resulted in high hole concentrations. Furthermore high hole mobilities ranging from 70–150 cm²/Vs were demonstrated in the Be-O codoped GaN samples with hole concentrations of $1-5\times10^{18}$ cm⁻³ [17]. At the same time, the semiconductor became degenerate for Be-O codoping. [17]. Somewhat smaller hole mobilities of 17 [6] and 40 cm²/Vs [19] were observed for Mg-O codoped GaN layers for similar hole concentrations as it can seen in Table 1. A continuous decrease in the acceptor ionisation energy with codoping was demonstrated in Mg-O codoping studies [6,19]. These results suggest that screening plays an important role in determining the electronic properties of codoped GaN.

The behaviour of GaN codoped with Mg and Si is more complex [18,20]. In studies of MOVPE grown material, the hole concentration first decreased by a factor of five with the addition of silane (SiH₄) dopant gas as would be expected for compensation with shallow donors [20]. How-

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Codoping pair	Growth method	[A]	[D]	T (°C)	p/n (cm ⁻³)	$\mu (cm^2/Vs)$	Ref.
		Be (cm ⁻³)	H ₂ O (Torr)				
GaN:Be-O	MBE	10 ²⁰	>3×10 ⁻¹⁰	650	5×10 ¹⁸	70	[17]
GaN:Be-O	MBE	5×10 ²⁰	>3×10 ⁻¹⁰	650	1×10^{18}	150	[17]
		Cp ₂ Mg/Ga	SiH ₄ (nmol/min)				
GaN:Mg-Si	MOCVD	7.6×10 ⁻³	0.11	1080	4.3×10 ¹⁷	11.5	[20]
		Cp ₂ Mg (nmol/min)	DEZn (nmol/min)				
GaN:Mg-Zn	MOCVD	0.7	0.616	1080	8.5×10^{17}	10.5	[20]
		2	2				
		Mg (cm $^{-3}$)	$O(cm^{-3})$				
GaN:Mg-O	Bulk solution	$10^{19}(0.1-0.5\%)$	$10^{18} - 10^{19}$	1400-1700	*	*	[16]
GaN:Mg-O	MBE	1.6×10^{20}	4×10^{18}	780	2×10^{18}	40	[19]
AlGaN:Mg-O	MBE	1.6×10 ²⁰	4×10^{18}	780	2×10^{18}	8	[19]
		Cp ₂ Mg (µmol/min)	O ₂ (ppm)				
GaN:Mg-O	MOCVD	0.36	15	1060	2×10^{18}	17	[6]
GaN:Mg	MOCVD ⁺	_	O ₂ /N ₂ -1%	1050	3×10 ¹⁸	2.6	[27]

Table 1. Reported experimental data for codoping experiments in GaN/AlGaN.

* semi-insulating samples were obtained.

⁺ the samples were annealed in N₂/O₂ mixture at 850°C for 20 minutes.

ever, at higher SiH₄ concentrations, the hole concentration increased by a factor of four. The authors attributed the observed behaviour to the competitive adsorption between Mg and Si on the same lattice site. Nakamura *et al.*, however, observed that hole concentration increased from 5×10^{17} to 5×10^{19} cm⁻³ in Mg doped GaN when Si concentration in the films increased in the range of 10^{16} – 10^{20} cm⁻³ [18]. The high net hole concentrations have not been observed by others in codoped GaN.

In this paper the relevant theory of codoping of widegap semiconductors is reviewed. We evaluate recent experiments of codoped GaN in light of recent theory.

2. Theory

The theory for codoping was developed more than forty-five years ago by Reiss *et al.* for elemental semiconductors using solution theory [12–13]. Codoping can lead to increased conductivity due to a number of factors including:

- enhanced equilibrium solubility,
- lowering of the ionisation energies,
- change in carrier mobility.

All three are operable in GaN.

2.1. Equilibrium phase relations

Impurity solubility can be affected by both changing the Fermi level and by ion pair formation. In the case of the effect of the Fermi level on solubility, the solubility of an acceptor is given by [12]

$$\frac{N_A}{N_{Ai}} = \exp\left[\frac{-(E_{Fi} - E_F)}{kT}\right]$$
(1)

where N_A and N_{Ai} are the acceptor concentrations for the doped semiconductor and the intrinsic semiconductor, respectively and E_F is the Fermi level. E_{Fi} is the intrinsic Fermi level. As it can be seen, there is an exponential dependence of acceptor concentration on the Fermi level. A similar expression can be written for donors.

For a wide range of acceptor concentrations the dependence of N_A on donor concentration, N_D , is given by

$$N_{A} = \frac{N_{D}}{2} + \sqrt{\left[\left(\frac{N_{D}}{2}\right)^{2} + N_{Ao}^{2}\right]}$$
(2)

where N_{Ao} is the solubility of acceptors at a low compensation ratio [12,13].

2.2. Pair formation

By forming stable ion pairs the solubility will also increase. For example, for Mg and O doping of GaN ion pairs will form according to the reaction

$$Mg^{-} + O^{+} = [Mg^{-}O^{+}]$$
(3)

Through the law of mass action the equilibrium constant is

$$K' = \frac{a_{MgO}}{a_{Mg^-} \cdot a_{O^+}},$$
 (4)

where a_i are the respective activities of the ions and ion pairs. In the case of a dilute solution the pair concentration P is given by

$$\Omega = \frac{P}{(N_{Mg} - P)(N_O - P)},\tag{5}$$

where Ω is the constant that depends upon the Coulombic interaction energy

$$\Omega = \frac{1}{2N} \exp\left(\frac{-\varepsilon}{kT}\right) \tag{6}$$

where N is the total site density (8.8×10²² cm⁻³ in GaN) and ε is the Coulomb pair energy [21]. This energy is given by

$$\varepsilon = \frac{-e^2}{\kappa}$$
(7)

where κ is the dielectric constant and *r* is the ion pair distance. For GaN, ε is approximately equal to 776 meV [15] for nearest neighbour pairs, where r = 1.95 Å (ignoring relaxation) and $\kappa = 9.5$ [22]. Using typical values for codoping ($N_{Mg} = 4 \times 10^{19}$ cm⁻³, $N_O = 5 \times 10^{18}$ cm⁻³), the calculated pair concentration is 9×10^{17} cm⁻³ at 1300 K.

Enhanced solubility due to ion pair formation at the growth temperature, 1300 K would be approximately equal to $e^{e/kT} = 10^3$. Figure 1 shows the experimental dependence of N_A on N_D for codoped GaN and the calculated dependence using Eq. (2). Included is the data for deliberately codoped GaN. In this case, Si was the donor, and Mg was the acceptor, and the concentration of each species was determined by SIMS. As it can be seen, the Mg concentration is independent of the donor concentration over the range studied. Thus, solubility enhancement is not observed in this range. This is consistent with the Reiss model [12,13]. Donor concentrations in excess of 2×10^{19} cm⁻³ would be needed in order to enhance the Mg solubility.



Fig. 1. Effect of codoping on the solubility of Mg in GaN. SIMS data is plotted for GaN:Mg-Si (squares). The data is fit to Eq. (2), and N_{Ao} is found to be 2.2×10¹⁹ cm⁻³ for GaN:Mg-Si. The dashed lines are calculated curves for values of N_{Ao} equal to 1×10¹⁸ (i), 110¹⁹ (ii), and 1×10²⁰ cm⁻³ (iii).

2.3. ADA complex formation

While neutral pair formation would increase the solubility of both acceptors and donors, Katayama-Yoshida et al. have proposed that formation of a charged nearest neighbour acceptor complex consisting of two acceptors and one donor (ADA complex) can lead to increased p-type conductivity in codoped GaN [15]. Maximum enhancement of the hole concentration is expected when an ADA complex is formed. The increased hole concentration is attributed to the low ionisation energy of the complex as compared to that of the simple substitutional acceptor [12,13,15]. An ionisation energy as low as 0 meV is predicted for this defect complex [15]. To calculate the effect of ADA complex formation on hole concentration, two types of acceptors are assumed for this model. The first type is formed by an ADA complex and has a concentration equal to $2N_D$, while the other is the isolated acceptor species with a concentration N_A -2 N_D , where N_A is the total acceptor concentration. This model assumes that every available donor will form an ADA complex. The activation energies of the ADA acceptor complex and the isolated acceptor are taken as 0.0 [15] and 200 meV [5], respectively. The expression for the hole concentration with one donor species and multiple acceptors is [23]

$$p + N_D = \sum_j \frac{N_{Aj}}{1 + pp_j} \tag{8}$$

where N_{Aj} is the concentration of acceptor species j, N_D is the donor concentration and $p_j = (g/N_v)exp(E_{Aj}/kT)$, with activation energy, E_{Aj} . For the calculations, the degeneracy factor is taken as g = 3.6 and the density of valence band states, $N_v = 1.8 \times 10^{19}$ cm⁻³ at 300 K [5].

The calculated hole concentrations for two cases, $N_A = 1 \times 10^{19}$ and 1×10^{20} cm⁻³ are shown in Fig. 2 (curves i and ii, respectively). An increase in the hole concentration with increasing N_D is observed. The hole concentration increases by an order of magnitude up to a level, $N_A = 2N_D$. Since the maximum concentration of the ADA complexes is fixed by the acceptor concentration, a decrease of hole concentration for $N_D > 0.5N_A$ is expected. For comparison, the simple compensation model [15] is also shown in Fig. 2. Clearly, the opposite behaviour is observed for the simple compensation model, shown in Fig. 2 (curves iii and iv) [15].



Fig. 2. Hole concentration as a function of N_D for the [ADA] model is shown with solid lines for $N_A = 1 \times 10^{19}$ (i) and 1×10^{20} cm⁻³ (ii). The optimum hole concentration where $N_A = 2N_D$ is plotted with diamonds. For comparison, the simple compensation model which assumes a single donor and (unpaired) acceptor is shown with the dashed lines for $N_A = 1 \times 10^{19}$ (iii) and 1×10^{20} cm⁻³ (iv). Finally, the random pair model is also plotted with dotted lines for $N_A = 1 \times 10^{19}$ (v) and 1×10^{20} cm⁻³ (vi).

Relative enhancement of the calculated hole density with codoping varied depending on the fixed acceptor concentration. The highest hole concentration is expected when $N_A = 2N_D$. This case with complete compensation is shown with open diamonds in Fig. 2. This limitation can be elevated by considering increased incorporation between A and D according to Reiss [12,13]. When incorporation of the dopant species varies according to Eq. (2), hole density increases along the line showed with open diamonds for the donor concentration $N_D > 0.5N_A$.

Formation of the nearest neighbour ADA complexes depends upon diffusion of donor and/or acceptors in the GaN matrix at the growth temperature. Therefore it is of interest to consider the interaction of donors and acceptors without close pairing, forming a random pair distribution. In the following discussion, it is assumed that the distance between D and A is $r = (3/4\pi N_t)^{1/3}$ [12], where $N_t = N_A + N_D$. The activation energy for the spatially separated ADA complexes is assumed to be given by $E_A = E_A^0 - e^2/2\kappa a$ [15]. Therefore as the ions become close to each other, the activation energy of the ADA complex decreases. Hole concentrations for the random distribution model as a function of donor concentration are calculated and are presented in Fig. 2. These calculations for random distribution of donors and acceptors with $N_t = 1 \times 10^{19}$ and 1×10^{20} cm⁻³ are shown (curves v and vi). It can be seen that hole concentration does not decrease as quickly as it does for the simple compensation model (dashed curves). However, only a small increase of p for $N_D \sim 5 \times 10^{19}$ cm⁻³ is observed.

To evaluate the effect of ADA complex formation on the hole concentration as a function of acceptor concentration, the hole density is calculated using Eq. (8) with a fixed donor concentration. These dependences are shown with solid lines for $N_D = 10^{18}$ and 10^{19} cm⁻³ in Fig. 3. Hole concentrations obtained using the simple compensation model with one donor, one acceptor and $E_A{}^0 = 200$ meV are also presented as dashed lines in the same figure for comparison. A square root increase of hole concentration is expected from the simple compensation model for high acceptor concentrations [5]. In contrast, a weak dependence of the hole density is observed for the ADA model at high acceptor concentrations.



Fig. 3. Calculated hole concentration as a function of N_A for $N_D = 1 \times 10^{18}$ and 1×10^{19} cm⁻³. The [ADA] and simple compensation models are shown with solid and dashed lines, respectively.

3. Comparison between theory and experiment

To determine the effect of oxygen codoping on carrier concentration for p-type GaN two sets of experiments were performed. The films for the first set were grown at a constant Mg dopant concentration in the gas phase, while the concentration of oxygen was varied from 0 to 80 ppm [6]. The room temperature carrier concentrations as a function of oxygen doping for this set are shown in Fig. 4(a). As it can be seen, the hole concentration of the codoped epilayers increased super-linearly upon oxygen doping. A hole concentration as high as 2×10^{18} cm⁻³ was achieved. For an oxygen concentration of ~30 ppm the conductivity type changes, however, from hole to electron conduction as indicated by a change in the sign of the Hall effect.

For the second set, the oxygen partial pressure during growth was kept constant at 4 ppm and the Mg dopant concentration was varied. The dependence of hole density on Mg flow rate for codoped samples is presented in Fig. 4(b). It can be seen that the hole concentration increased by more than an order of magnitude up to 1×10^{18} cm⁻³ and then saturated. For comparison, Mg-doping without oxygen grown under similar conditions produces p-type samples with a concentration of 2×10^{17} cm⁻³ and a resistivity of 3.5Ω cm.

There is some qualitative agreement between the observed hole dependence on dopant partial pressure and that predicted using the ADA complex model calculations. For example, an increase of hole density was observed experimentally with increasing donor dopant as seen in Fig. 4(a) as predicted by theory. Furthermore, the data presented in Fig. 4b indicates that the hole density is nearly fixed at a specific concentration by the donor concentration, N_D , which is agreement with theory. In Figs. 4(a) and 4(b), the solid line is calculated from Eq. (8) taking N_A and N_D , respectively, as a fitting parameter. For these calculations two acceptor energies were assumed with energies of zero and 0.20 eV. In Fig. 4(b), the value of $N_D = 1.3 \times 10^{18}$ cm⁻³ is used to fit the data, and there is good agreement between theory and experiment.

However, in Fig. 4(a), the increase in the experimental hole concentration is steeper than what is predicted by theory. In this case the fitting parameter N_A was equal to 1×10^{19} cm⁻³. The sharp increase in the measured hole concentration, could in part be due to other compensation mechanisms at low hole concentrations. It should be noted that the usual bell-like shape dependence of hole concentration on acceptor concentration often reported in GaN:Mg was not observed. This was previously attributed to the formation of compensating donor defects [5]. The ADA model does not include compensation by donor complexes such as $[Mg_{Ga}V_N]^{2+}$.

Experimental data, presented in Fig. 4(a) is for the heavy doping case ($p = 8 \times 10^{16}$ cm⁻³ was measured without oxygen present) with N_A on the right hand side of the bell-like shape maximum [6]. It is likely that oxygen substitutes on the V_N^{3+} sites. Thus, more Mg_{Ga} acceptors will become active upon codoping with oxygen since the $[Mg_{Ga}-V_N]^{2+}$ complex will not form, providing an additional increase of the hole density. The quenching of the 2.8 eV photoluminescence band observed with codoping was previously attributed to a decrease in compensating native donor defects [24,25].

While the ADA complex model qualitatively describes the aforementioned experimental codoping data, there still remain several unanswered questions. The first comes from



Fig. 4. Experimental carrier concentration as a function of oxygen concentration during growth (a). The solid line is obtained by plotting Eq. (8) with $N_A = 1 \times 10^{19}$ cm⁻³. In (b), the hole concentration is plotted as a function of the Cp₂Mg flow rate during growth. The oxygen flow rate was held constant at 4 ppm. The solid line is a fit of Eq. (8), with $N_D = 1.3 \times 10^{18}$ cm⁻³.

the fact that while a degenerate acceptor level $E_A \sim 0$ meV was utilised in the calculations, non-zero activation energies were measured for the samples shown in Fig. 4 [6] and also in Ref. 19. Nevertheless degenerate p-type GaN was observed in other studies [17,18] where the enhancement of hole densities by codoping was observed.

The different behaviour of Si codoping in GaN can be tentatively explained by the ADA model. The tendency towards ADA complex or pair formation depends on the chemical nature of the participating atoms. Pairs normally forming stable compounds are more likely to form com-

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plexes [12,13]. Thus Mg-O pairs are more likely to be formed in GaN than Mg-Si pairs. The independence of N_A on Si donor density is attributed to a donor concentration that is insufficient to produce significant enhanced incorporation of acceptors, as discussed earlier. There is also an obvious impediment for formation of nearest neighbour complexes (ADA), since both of these atoms substitute on the same (Ga) lattice site. Competitive adsorption also affects the mutual incorporation of Mg and Si. It may decrease the solubility limits of these dopants in GaN [20]. Similarly, the highest hole concentrations were obtained in GaN samples grown with a decreased gallium flow rate with respect to the optimum Ga-flux, since the formation energy of Mg_{Ga} increases with gallium chemical potential.

While the ADA model explains some of the observed codoping behaviour, the lack of low ionisation energy for acceptors in some codoping studies indicates other mechanisms are important. It has been proposed that screening by impurities may play an important role in codoping [12,13]. Thus a combination of several factors, including complex formation, decrease of compensation, increase in acceptor incorporation, and screening effects leading to a decrease in ionisation energy could all be responsible for the observed increases in hole concentration. It also should be noted that recently a potential fluctuation model was used to describe the observed increase of the hole concentration upon codoping [26]. It also predicted very weak temperature dependence of the hole concentration. Finally, a recent study of annealing of GaN:Mg in the presence of oxygen suggests that surface effects can play some part in the experimentally observed enhancement of hole concentrations and conductivities [27].

4. Conclusions

The fact that oxygen and silicon donors behave differently as codopants in GaN:Mg indicates that the observed codoping effects are not simply due to Fermi level effects. Oxygen addition has an appreciable effect on the hole concentration in GaN. There is experimental evidence that shallow acceptor complexes are forming from the measured dependence of hole concentration on Mg dopant concentration for oxygen codoped samples. Nevertheless codoping remains a complex phenomenon and several mechanisms are presumably operable.

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Invited paper

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- Organization of other topical sessions is not excluded, and depends on input from their organizers as well as on number and quality of submitted papers.

The Organizers of topical sessions are renowned domestic and international experts in relevant branches of science and technology. Particular topical sessions are filled with papers invited by Session Organizers and by presentations submitted by young researchers, M.Sc. and Ph.D. students. Supervisors or tutors of young researchers should recommend the submitted papers.

The main aim of the Symposium is to build a nation wide debating forum for young researchers and Ph.D. students with strong participation of experts and young research fellows from abroad. Membership of young scientists in IEEE (students and GOLD members) and SPIE will be emphasized during a special Society Evening and IEEE B-B-Q Reception.

The Symposium publications are subject to a standardized peer reviewing process, as in archival journals, and are printed in renowned series Proceedings of SPIE (www.spie.org and www.spie.pl) in English language. Some papers are also published in Elektronika Monthly, a journal of Association of Polish Electrical Engineers. Elektronika is a medial patron of the Symposium. The official language of the Symposium is English. The presentations are allowed also in Polish, Russian and German.

The Symposium Internet information site is: http://nms.ise.pw.edu.pl/ieee/sympozja.

The Symposium Organizers invite experts in relevant subject, embracing the widely understood Symposium scope, to declare organization of specialistic sessions.

The Symposium Organizers invite warmly students (Eng., M.Sc., Ph.D.), young researchers from academia, research institutions, innovative spin-offs and industry to submit papers and participate in WILGA meeting. WILGA is an unforgettable and irreplaceable experience for young people.

All information about the Symposium, organization problems, session proposal, paper submission, participation questions are available only through electronic way under the address: photonics@ise.pw.edu.pl; R.Romaniuk@spie.pl, R.Romaniuk@ieee.org.

The Symposium participation costs are just minimal. There is no fee. The only costs are accommodation in WILGA WUT Resort Centre. These costs are estimated for 2003 to be 55 Polish Złoty per day, for night and three meals a day. That is around \$14. The participants book the rooms in WILGA of their own, under the telephone number 0-prefix-25-685-30-17 (45,47). The information about the WILGA WUT Resort Centre is available on the web: http://www.info1.pl/Noclegi/osrodki/mazowieckie/wilga/start.htm