

Optically detected magnetic resonance of native defects in $\text{Al}_x\text{Ga}_{1-x}\text{As}$

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The structure and recombination properties of native defects in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ have been studied with use of optically detected magnetic resonance (ODMR). Three types of spectra were observed. Extended results are presented for the first type, which has been attributed to Ga interstitials. Spectra taken at different microwave frequencies suggest that alloy disorder affects the line shape. The dependencies on a variety of parameters in molecular-beam and organometallic-vapor-phase epitaxy are explored. Spectral dependence of the ODMR reveals that the $+2$ level of the interstitial is 0.56 ± 0.1 eV above the acceptor level. A brief comparison with other interstitials is presented. The second type of spectrum is sharp with a donorlike g value (1.947 ± 0.003). This ODMR line is attributed to Si donors by comparison to the electron-paramagnetic-resonance work of Bottcher *et al.* [Phys. Status Solidi B **58**, K23 (1973)]. The third type of spectrum has an acceptorlike g value (2.183 ± 0.009 for \mathbf{B} parallel to $[110]$). It may be due to a transition-metal impurity or an Al or Ga antisite.

I. INTRODUCTION

$\text{Al}_x\text{Ga}_{1-x}\text{As}$ is currently very interesting because it is closely lattice matched to GaAs and can be used in forming simple heterostructures and an endless variety of more complicated device structures. However, the quality of the $\text{Al}_x\text{Ga}_{1-x}\text{As}$ material is not as good as that of GaAs and efforts are underway to improve it. Specifically, oxygen and carbon are believed to be important residual native defects in both molecular-beam-epitaxy (MBE) and organometallic-vapor-phase-epitaxy (OMVPE) material. Intrinsic defects, such as vacancies, interstitials, and antisites are also imperfections which, if present in sufficient concentration, would produce deleterious effects on the final electrical and optical properties. Since it is a ternary alloy, more different intrinsic defects are possible in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ than in a simple binary (GaAs) or an elemental (Si) semiconductor. Variable band gap and alloy disorder add new interest to the study of defects in $\text{Al}_x\text{Ga}_{1-x}\text{As}$.

Optically detected magnetic resonance (ODMR) has recently been applied to study the identity and structure of native defects in MBE-grown $\text{Al}_x\text{Ga}_{1-x}\text{As}$. Two aspects of the technique are crucial to this work. First, the magnetic resonance provides the detailed atomic structure of the defect as in electron paramagnetic resonance. Second, the ODMR is uniquely suited to the thin epitaxial layers since the resonance is detected on photoluminescence. In previous papers, the Ga interstitial was identified through hyperfine interactions in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ grown at lower than optimal temperatures¹ and some of the variations with growth parameters are described.² The purpose of the present paper is to present extended results on the Ga interstitial and a brief account of the other resonances observed in the course of studying the interstitial.

The results are presented as follows. A description of the sample growth techniques and ODMR spectrometers is given in Sec. II. Results on a set of $\text{Al}_x\text{Ga}_{1-x}\text{As}$ samples with different alloy composition x , growth temperature, doping, and growth technique are given in Sec. III. These lead to a picture of a family of interstitial defects and provide a measurement of an energy level. A distinct donor ODMR is described in Sec. IV and attributed to Si donors through comparisons with other work. A third type of ODMR line is reported in Sec. V and deep-acceptor models are discussed. Section VI provides a conclusion.

II. EXPERIMENTAL ASPECTS

Most of the work described herein was done on a set of samples grown by MBE on semi-insulating GaAs substrates in a Varian Associates Model GEN 1.5 machine. ODMR was stronger for samples grown at lower than optimal substrate temperature. These samples often exhibited high resistivity at room temperature. Their near-band-edge photoluminescence at 1.6 K was weak and broad but usually allowed a determination of an approximate Al mole fraction (x). Their deep photoluminescence was strong and nearly featureless in the energy range from 0.75 to 1.1 eV.¹ This type of luminescence provides an ideal starting point for ODMR of deep centers.

A few samples grown by OMVPE on semi-insulating GaAs were studied. These were weakly p type presumably due to carbon acceptors. The OMVPE and MBE samples had similar photoluminescence properties.

The ODMR was detected as variations in the photoluminescence intensity coherent with chopped microwave sources. Most of the data were taken in Voigt geometry in a spectrometer consisting of an optical Dewar in a 9-

in. electromagnet with 24-GHz microwaves. The frequency dependence was obtained using an Oxford Instruments 6.5-T Spectromag system with 35-GHz microwaves. Photoluminescence was excited either by a HeNe laser or a Kr⁺ laser with power to the sample in the range from 3 to 50 mW. The sample temperature was from 1.5 to 1.6 K. Depending on the spectral range, the luminescence was detected by either an EG&G Si photodiode, a North Coast cooled Ge photodiode, or a Judson cooled InSb photodiode. Signal averaging was often necessary to obtain good signal-to-noise ratio.

III. Ga INTERSTITIAL AND RELATED SPECTRA

A. Microwave frequency dependence

The previous communication focused on the spectrum of a Si-doped sample at 24 GHz (Ref. 1) which is resolved into four lines and is nearly isotropic. The outer two lines are broader and have less amplitude than the inner lines, indicating that the spectrum is due to a defect with a nucleus which has two nuclear-spin $I = \frac{3}{2}$ isotopes. Fitting the spectrum shows that it can be attributed to the ⁶⁹Ga and ⁷¹Ga moments and abundances with the parameters $g = 2.025 \pm 0.006$ and $A(^{69}\text{Ga}) = 0.050 \pm 0.001 \text{ cm}^{-1}$ for B near [001]. The magnitude of the hyperfine coupling A indicates a state of A_1 symmetry which current theories predict for only the interstitial among Ga defects.³⁻⁵ In the first paper the slight anisotropy was attributed to pairing of the interstitial with a second defect. Here we examine this issue more carefully by studying the microwave frequency dependence.

The angular dependence of the 35-GHz ODMR in Si-doped Al_xGa_{1-x}As is shown in Fig. 1. As at 24 GHz,

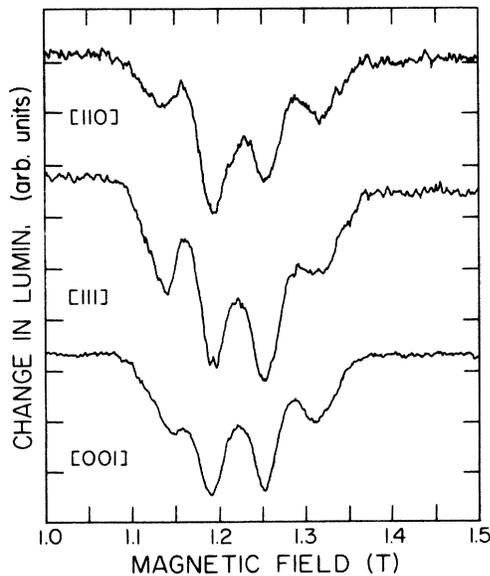


FIG. 1. Angular dependence of the ODMR at 34.6 GHz and 1.5 K in sample no. 5 (MBE88). The spectrum, designated as (AlGaAs-) NRL-1, is nearly isotropic and exhibits the four-line structure attributed to ⁶⁹Ga and ⁷¹Ga hyperfine structure.

the spectrum is partially resolved into four lines with some anisotropy. A comparison with the data at 24 GHz (Ref. 1) shows differences in the spectra for the [110] and [111] spectra with close similarity for the [001] direction. The simplest interpretation of this result is a distortion along the [111] direction producing an anisotropy in g values. Since all possible {111} directions are equivalent when the field is in a [001] direction, the spectrum for this direction would have a single g value and be invariant with microwave frequency. Attempts to make a quantitative analysis from this starting point have failed so far which indicates that the symmetry is probably lower than [111].

Some understanding of the symmetry can be gained by examination of the Ga interstitial site in Al_xGa_{1-x}As (see Fig. 2). Charge-state^{1,4} and simple ionic bonding considerations place the Ga_i at the tetrahedral interstitial site surrounded by four As atoms. As comparison with the results in OMVPE samples will show, the shell nearest the interstitial has all four As atoms in Si-doped MBE samples. The second shell consists of six atoms of Al and Ga if no impurities are present. This shell is only 15% farther from the interstitial than the first shell and thus can have a significant effect on the resonance parameters. Alloy disorder is probably important for interstitials at any site. Thus the slight asymmetry is attributed to the alloy disorder of the second shell with the possibility of an impurity in that shell as well.

B. Al-fraction, substrate-temperature, and doping dependencies

ODMR has been studied in a set of samples encompassing a range of growth parameters (see Table I). The Ga_i spectrum is favored in samples grown at low-substrate temperatures with Al fractions above 10%. A broad positive ODMR is observed in MBE-grown GaAs and, as the Al fraction increases, the negative interstitial signal develops. Al fractions greater than 45% have not yet been explored. When higher substrate temperatures are employed, no interstitials are detected.

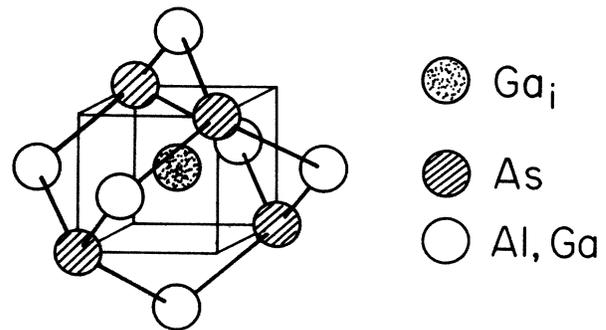


FIG. 2. The atomic structure of the Ga_i defect at the tetrahedral interstitial site surrounded by four As atoms. The interstitial is surrounded by an octahedron of Al and Ga atoms at a distance only 15% greater than the As atoms.

TABLE I. Observations of ODMR in samples grown by MBE. NRL denotes Naval Research Laboratory, ND denotes not detected.

Sample	T_{sub} (°C)	x	Doping (cm^{-3})	Ga_i NRL-1	Donor NRL-5	Deep acceptor NRL-6
No. 1 (MBE91)	620	0.06	Si, 3×10^{16}	ND	ND	
No. 2 (MBE159)	570	0.1	Si, 3×10^{16}	weak	ND	
No. 3 (MBE104)	700	0.10	Si, 1×10^{17}	ND	ND	
No. 4 (MBE92)	620	0.22	Si, 3×10^{16}	moderate	ND	ND
No. 5 (MBE88)	620	0.24	Si, 3×10^{16}	strong	ND	
No. 6 (MBE89)	620	0.39	Si, 3×10^{16}	weak	strong	
No. 7 (MBE103)	700	0.30	Si, 1×10^{16}	ND	ND	
No. 8 (MBE93)	620	0.45	Si, 3×10^{16}	weak	strong	
No. 9 (MBE156)	620	0.45	Si, 1×10^{17}	strong	moderate	
No. 10 (MBE118)	655	0.2	Be, 3×10^{17}	strong	ND	strong
No. 11 (MBE148)	620	0.25	Be, 1×10^{18}	ND	ND	
No. 12 (MBE161)	620	0.35	undoped	weak	ND	

The same Ga_i is found both in undoped and Be-doped samples. Thus there is not a dopant which pairs closely enough to alter the spectrum in the lightly doped MBE samples. A heavily-Be-doped sample exhibits a positive ODMR spectrum split into four lines with a hyperfine constant of about 0.069 cm^{-1} . (The spectra are listed in Table II.) Due to poor resolution this spectrum cannot be definitely assigned to Ga. ^{75}As is a second possibility since As_{Ga} antisites with similar hyperfine constants have been reported in GaAs.^{6,7} An undoped sample exhibited a weak Ga_i spectrum, which requires much slower than normal microwave chopping for optimal detection. This probably reflects slower processes due to greater average distance between emitting and capturing centers.

C. Spectral dependence

Since the Ga interstitial is a deep donor, the lightly-Be-doped sample no. 10 (MBE118) was chosen as a likely sample for detecting the positive donor-acceptor ODMR process. The spectral dependence was studied using

filters which passed a range of a few tenths of an eV as the signals are fairly weak. Often the deep luminescence bands are this wide in energy. The Ga_i signal is negative from the band-gap energy down to 0.70 eV but positive in the energy range from 0.44 to 0.69 eV (see Fig. 3). Further confirming results in a Si-doped sample will be presented in Sec. IV. The data also reveal a new positive ODMR at both the high- and low-energy ends, which will be discussed further in Sec. V.

A consistent picture of the Ga_i ODMR processes can be constructed (see Fig. 4) and an energy level deduced. The higher-energy, negative ODMR is observed when spin flips at the Ga_i^{2+} allow increased nonradiative electron capture and decrease the observed emission. If this capture is the rate-limiting step, then the positive ODMR is observed when the lower-energy radiative recombination process is detected. Strong electron capture is consistent with the double positive-charge state of the interstitial. The positive ODMR reveals that the $+ / 2 +$ energy level is about $0.56 \pm 0.1 \text{ eV}$ above the acceptor level in $\text{Al}_{0.2}\text{Ga}_{0.8}\text{As}$. This value is consistent with the prediction of 0.5 eV above the valence band in GaAs.⁴

TABLE II. $\text{Al}_x\text{Ga}_{1-x}\text{As}$ ODMR spectra.

Spectrum (AlGaAs-)	Growth Technique	Linewidth (mT)	g	Nuclear spin	Hyperfine constant (cm^{-1})	Positive ODMR range (eV)	Defect model
NRL-1	MBE	22	2.025	$\frac{3}{2}$	0.050/0.064 [001]	0.44—0.69	Ga_i
NRL-2	MBE (MBE148)			$\frac{3}{2}$	0.069	0.70—1.1	Ga_i or As_{III}
NRL-3	OMCVD			$\frac{3}{2}$	0.052	0.70—1.1	Ga_i pair
NRL-4	OMCVD			$\frac{3}{2}$	0.058	0.70—1.1	Ga_i pair
NRL-5	MBE	12.5	1.947	0		0.70—0.95	Si_{III}
NRL-6	MBE	35	2.183	0		0.44—0.69 1.2—1.8	Al_{As} , Ga_{As} , or Fe
			[110]				

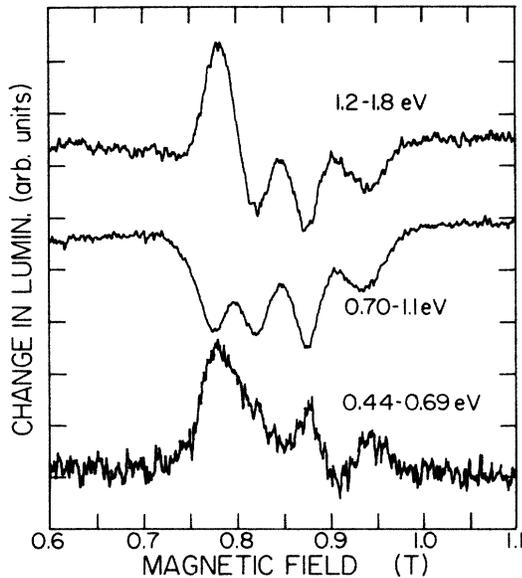


FIG. 3. Spectral dependence of the ODMR in a Be-doped sample, no. 10 (MBE118). A new spectrum (NRL-6) interferes with the low-field lines of the Ga_i spectrum at the lowest and highest energy. The Ga_i spectrum is positive in the (0.44–0.69)-eV range.

D. Annealing

Two samples which exhibited strong Ga_i signals were annealed for 30 min at 850°C in an As overpressure. In each case the (0.7–1.1)-eV photoluminescence increased but the Ga_i ODMR decreased. Since capture at Ga_i competes with emission in this range, both effects are consistent with a decrease in the number of interstitials. Thus, some Ga interstitials are removed at 850°C. Since transition metals may diffuse from the substrate to the interface at these temperatures, some care must be taken in doing and interpreting annealing experiments.

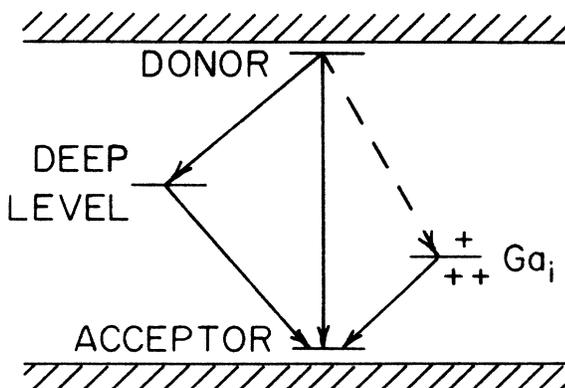


FIG. 4. Recombination processes involving the Ga interstitial. Nonradiative capture by Ga_i causes decreases at resonance for the near-band-edge to midgap luminescence. Positive signals are observed for the low-energy Ga_i -to-acceptor process.

E. Samples grown by OMVPE

A few samples grown by OMVPE were studied and two exhibited strong ODMR signals (see Fig. 5). The spectra are very weakly split into four lines with a hyperfine constant about equal to that of the Ga_i spectrum in MBE samples. Unlike the ODMR in MBE samples, the spectra are positive in the (0.7–1.1)-eV range. Sample 13 (OM214) was weakly *p* type, although not intentionally doped. Carbon is expected to be the dominant impurity in this sample and nearest-neighbor pairing with the interstitial ($C_{As}-Ga_i$) may be responsible for the different ODMR spectrum and recombination energy. Sample 14 (OM309) was undoped and had high resistivity. Si and O contamination may be present in this sample due to a fresh trimethyl Al source. The reason for the better-resolved hyperfine spectrum in Sample 14 (OM309) is unclear. Although more study of these samples is required, the results in OMVPE samples show that some residual impurities form nearest-neighbor pairs with interstitials causing changes in their resonance parameters and transition energies.

F. Discussion of Ga interstitials

Further experiments have provided new information on the atomic structure, occurrence, recombination properties, and electronic energy level of the Ga interstitial in $Al_xGa_{1-x}As$. A fairly complete and consistent picture has emerged. It is interesting to compare these results with work on closely related interstitials in other hosts (see Table III). Interstitials have been detected in B_2O_3 -doped GaP (Ref. 8) and electron-irradiated Si:Ga (Ref. 9) and ZnSe.¹⁰ The stability of these interstitials varies wide-

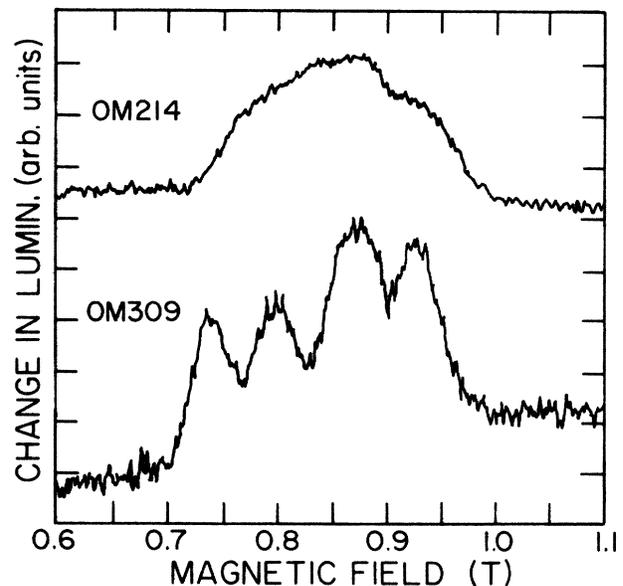


FIG. 5. ODMR at 24.1 GHz and 1.6 K for samples grown by OMVPE. The field direction is [110]. The top spectrum (NRL-3) is barely showing a four-line structure, while the bottom spectrum (NRL-4) is nearly as well resolved as the spectrum in MBE samples.

TABLE III. Interstitials in semiconductor hosts.

Interstitial	Host	Growth prep.	Stability (K)	Localization (%)	Author
Ga^{2+}	$\text{Al}_x\text{Ga}_{1-x}\text{As}$	MBE		18	This work
Ga^{2+}	GaP	OMCVD		26	Lee ^a
$\text{Ga}^{2+}-\text{Ga}_{\text{Si}}^-$	Si	electron irradiated	470	50	Watkins ^b
Zn^+	ZnSe	electron irradiated	220	60	Rong and Watkins ^c

^aReference 8.^bReference 9.^cReference 10.

ly. The degree of localization of each interstitial wave function can be expressed by determining the ratio of the experimental central hyperfine constant to that calculated for the corresponding free ion.¹¹ The Ga interstitial in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ is the least localized of the set. More work is necessary to achieve an understanding of these trends.

IV. DONOR LINE

A. Experimental aspects

A sharp unsplit ODMR is observed in the samples with the highest Al fraction (see Table I). In sample no. 6 (MBE89), the signal is negative with $g=1.947$ and a linewidth of 12.5 mT. Since the spectrum is sharp with a g value smaller than the free-electron value, the spectrum arises from a donor which is either an unintentional impurity or the dopant Si. The resonance parameters vary somewhat from sample to sample and thus may depend on x and possibly defect concentration.

This spectrum has a different dependence on sample parameters from Ga_i . It has not been observed in samples with Al fractions of less than 0.39. A striking change took place on 850°C annealing of a Si-doped, high- x sample (no. 9, MBE156, see Fig. 6). While the interstitial signal decreased, the donor signal changed from negative to positive.

Since a positive signal indicates that the ODMR is directly in the recombination cycle of the defect, the spectral dependence of sample no. 9 (MBE156) was studied (see Fig. 7). The sharp line is associated with the emission in the range from 0.70 to 0.95 eV. Note that the Ga_i ODMR becomes positive in the (0.44–0.69)-eV range as in the Be-doped sample (Fig. 3).

B. Assignment to Si

In the absence of resolved hyperfine interactions, magnetic-resonance spectra must be assigned to defect structures through correlations. The sharp donor line reported here is first compared with results for oxygen impurities.

The presence of oxygen in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ grown by MBE has been detected by secondary-ion-mass spectro-

scopy (SIMS) and correlated with low substrate temperature during growth¹² and contamination from fresh sources.¹³ Its presence in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ produces high resistivity.¹⁴ These features correlate with the present data. The resonance parameters for the sharp donor line are very similar to the g value of 1.996 and linewidth of 15 mT for O_p in GaP.¹⁵ These values were obtained from an EPR study of Ga_2O_3 -doped bulk GaP. The O_p line often shows up in ODMR studies of GaP.⁸ A comparison of the energy of the ODMR with published results for oxygen photoluminescence in GaAs (Ref. 16) and its alloys (Refs. 17 and 18) give further evidence that the sharp donor line is oxygen.

However, a better correlation exists between the sharp donor ODMR and an EPR line in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ with x ranging from 0.7 to 0.85.¹⁹ The EPR has a g value of 1.963 ± 0.002 with linewidths from 2.6 and 4.0 mT in different samples. The assignment of this EPR to donors is based on a correlation between the strength of the reso-

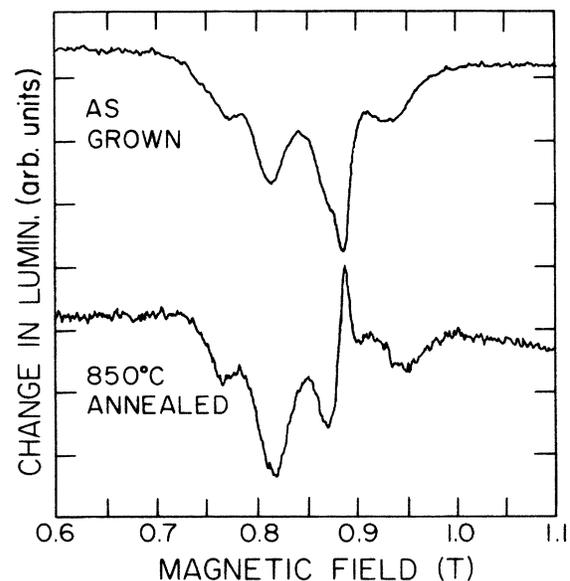


FIG. 6. ODMR before and after annealing in sample no. 9 (MBE156). The gain is increased for the annealed sample. The sharp single line (NRL-5) reverses sign upon annealing.

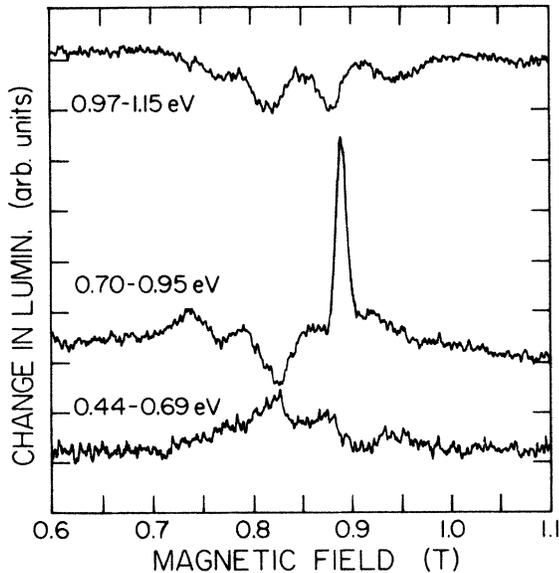


FIG. 7. Spectral dependence of the ODMR in sample no. 9 (MBE156) after annealing. The sharp line, attributed to Si on the III lattice site, is on emission in the (0.70–0.95)-eV range.

nance and the carrier concentration. The g value is very close to that of the sharp ODMR. The ODMR linewidth is larger as is usually the case due to donor-acceptor exchange interaction.²⁰ Based on this correlation, the sharp donor line is assigned to Si on the III lattice site.

Other aspects of the present results are consistent with this assignment. The line has only been observed in Si-doped samples. The observation for x greater than 0.39 suggests that the resonance is associated with the X conduction-band minimum and the g value is similar to other donor g values for indirect-gap semiconductors. The existence of both positive and negative signals is consistent with initial capture into Si-donor states preceding all the deep luminescence processes (see Fig. 4).

V. DEEP-ACCEPTOR LINE

A. Experimental aspects

A broad, positive ODMR was observed in the Be-doped sample no. 10 (MBE118) (see Fig. 3). It has a g value of 2.183 and a linewidth of 35 mT for B parallel to [110]. This width is quite broad for a line not exhibiting any structure and could either be due to a weak central hyperfine interaction or strong ligand interactions. Thus the defect may be intrinsic and involve Al, Ga, or As. The large positive g shift suggests an acceptor. The line exhibits a slight anisotropy moving from 784 mT for [110] to 798 mT for [112].

The spectral dependence is quite interesting (see Fig. 3). Positive signals are observed in two energy ranges: from 0.44 to 0.69 eV and from 1.1 eV to the band edge (1.8 eV). Apparently both the capture and emission at this center are radiative. The spin dependence of either process will produce positive ODMR for both. Since the g shift suggests an acceptor, it is natural to place the level in the lower half of the gap.

B. Possible defect models

Two defect models seem possible in the light of the present data. The line may be due to a transition-metal impurity, possible in the $3d^7$ state. Indeed, Fe^+ , Co^{2+} , and Ni^{3+} have g values around 2.2 in III-V hosts.²¹ However, the linewidths are considerably narrower than the 35 mT observed here and the slight anisotropy differs from that expected for the $S = \frac{3}{2}$ ions.²² An alternate assignment is the Al or Ga antisite. These would also be deep acceptors. A spectrum observed in bulk-grown GaAs has been tentatively attributed to a $\text{Ga}_{\text{As}}\text{-B}_{\text{Ga}}$ pair.²³ This EPR spectrum has resolved g anisotropy with $g_{\parallel} = 2.11$ and $g_{\perp} = 2.89$. The linewidth is 61 mT at 2 K. Again the parameters are similar but not really close to those observed in $\text{Al}_x\text{Ga}_{1-x}\text{As}$. Al or Ga antisites but no definite conclusions can be drawn at the present time.

VI. SUMMARY

Variety and detail have emerged in the ODMR study of deep defects in $\text{Al}_x\text{Ga}_{1-x}\text{As}$ by varying growth and observation parameters. An energy level was measured for the Ga interstitial and a family of similar defects found. An ODMR due to Si donors was detected for high- x values. A deep-acceptor resonance was also found in a Be-doped sample.

These results illustrate the power of ODMR detected through photoluminescence to study deep centers in $\text{Al}_x\text{Ga}_{1-x}\text{As}$. The sensitivity of the technique is excellent. Due to the high spins of the host nuclei, the resolution for intrinsic defects is barely adequate and could be improved by nuclear double-resonance techniques, such as optically detected electron-nuclear double resonance (ODENDOR).

This work brings closer certain new possibilities. ODMR might contribute to the understanding of the anomalous properties of donors in $\text{Al}_x\text{Ga}_{1-x}\text{As}$, the DX problem. Ion implantation of III-V materials could be studied both before and after annealing. Since the method is possible wherever photoluminescence is detected, it can also be applied to defects at or near interfaces or in confined layers. Lateral resolution is possible through scanning and focusing of the exciting laser beam.

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