Identification of residual donors in high-purity epitaxial GaAs with the use of magneto-optical spectroscopy

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We report the first identification of residual donors in high-purity, vapor-phase epitaxial GaAs using the photoluminescence technique in the presence of applied magnetic fields. Identifications were made from complexes of the neutral-donor—bound-exciton type in which the transitions occurred between excited initial and final states of the complex. The same residual donors were identified from ionized-donor—bound-exciton complexes in the same crystal. These measurements were also made in applied magnetic fields. The magnetic field compresses the wave function which sharpens the optical transitions. It also separates the different donors when viewed from neutral-donor—bound-exciton transitions. The magnetic field separation of different donors, when viewed from the ionized-donor—bound-exciton transitions, was not significant in fields up to 40 kG. An exchange energy of $\sim 30 \mu$V was measured from the magnetic field splitting of the ionized-donor—bound-exciton π lines.

I. INTRODUCTION

The interest in GaAs as a semiconductor material for technical applications has been steadily increasing in recent years. High-quality material is important for many of these applications. To improve the quality requires a knowledge of the residual impurities in undoped, "high-purity" material. The acceptors, having relatively large binding energies as compared to the donors (~ 30 vs 5.7 meV), can be identified quite easily. The shallow hydrogenic donors, on the other hand, have small binding energies and also have small central-cell corrections. This makes the resolution of different donors resulting from different chemical impurities difficult to achieve. The early experiments from which different chemical donors were identified employed high-resolution, Fourier-transform infrared magnetospectroscopy (FTIR) which used the modulated photoconductivity detection technique to monitor the $1S$-$2P_{-1}$ transition in a fixed magnetic field. The first experiments were done by Stillman et al.[1] using high-purity, vapor-phase epitaxial (VPE) GaAs, while Stradling et al.[2] performed similar experiments using liquid-phase epitaxial material. The donors in these experiments were residual donors. Subsequent experiments by Wolfe and co-workers,[3,4] which used the same measurement techniques on back-doped samples, permitted them to assign chemical identification to some of the residual donors.

More recently, shallow residual donors have been identified in high-purity VPE GaAs using high-resolution photoluminescence spectroscopy.[5,6] The optical transitions that were used to identify the residual donors result from the collapse of neutral-donor—bound excitons. The decay of an exciton bound to a donor (acceptor) may leave the donor (acceptor) in an excited state. This was first pointed out by Thomas and Hopfield.[7] They observed transitions in CdS that were characterized by large magnetic field splittings and negative diamagnetic shifts which they tentatively identified with transitions of this type. Excited-

state transitions of this type were later identified in GaP,[8] CdSe,[9,10] CdS,[11] ZnO,[12] and ZnSe.[13] Residual donors have subsequently been resolved in GaAs by Almassy et al.[5] from optical transitions resulting from the collapse of an exciton bound to an excited donor state, leaving an excited terminal state. The terminal state of this transition, from which chemical identifications are made, is the $n = 2$ state. When the terminal state is a $2S$ state, then the central-cell correction to this state is assumed to be $\frac{1}{8}$ of its value for the $1S$ state. In transitions of this type it is possible to observe $\frac{7}{8}$ of the total central-cell correction. When the terminal state is a $2P$ state the full central-cell correction is observed.

Emission lines have been observed on the high-energy side of the neutral-donor—bound-exciton transition (D^0,X) in many materials, CdTe,[14] GaAs,[15,16] CdS,[17] and ZnSe.[13] These transitions were interpreted as excited states of the D^0,X but with very little detail as to the nature of the excited states. Guillaume and Lavallard[18] proposed a rigid-rotator model to explain these excited states in CdTe. In this model the hole is excited to rotate around the fixed donor analogous to rotation of diatomic molecules. This model had difficulty in predicting the observed energies for the excited-state transitions. A non-rigid-rotator model was subsequently proposed by Ruhle and Klingenstein,[19] which was successful in predicting the excited-state energies in InP and GaAs. It was the collapse of excitons bound to the $n = 2$ rotational state of the donors from which the residual donors were resolved in Ref. 5. It has been observed that in many crystals the D^0,X state as well as the first non-rigid-rotational state are broadened while other bound-exciton states are not. Herzberg and Spinks[20] have observed broadening of rotational lines from diatomic molecules and have also observed that this broadening decreased with increasing rotational quantum number. It has been proposed[21] that an analogous broadening mechanism is responsible for the broadening of the D^0,X transition as well as the transition from the first non-rigid-rotational state. It was observed that when

the ground state was broadened the excited- ($n=2$) state transitions were also broadened. The energy separation between the D^0,X state and the first rotational state in GaAs is $\sim 50\,\mu$V. If one attempts to resolve residual donors from the excited-state ($n=2$) transitions of these two states, the combined line broadening and small energy separation render it impossible in most cases. Almassy et al.[5] circumvented this problem by observing the $n=2$ states resulting from the collapse of the exciton bound to the second non-rigid-rotational state of the donor, which is not broadened. This has the advantage that the experiment is done in zero magnetic field and therefore the donor energies are directly measured. The scheme has the disadvantage that the intensity of the $n=2$ state associated with the second non-rigid-rotational state is considerably less than the intensity of the $n=2$ state associated with the first non-rigid-rotational state.

In this paper we report the first identification of residual donors in GaAs from the transition involving an exciton bound to the first non-rigid-rotational state. The terminal state consists of the excited state ($n=2$) of the electron on the donor. The observation of different residual donor species from this transition is made possible by performing the experiment in a magnetic field. The magnetic field produces two effects: (a) It separates out states with different orbital angular momentum and (b) it compresses the wave function which sharpens the lines and separates the donors. In the final state the transition can terminate in either the $2S$ or $2P$ state. From parity arguments it can be shown that the initial state of the D^0,X transition has odd parity. The $2S$ final state in this transition will have even parity whereas the $2P$ final state will have odd parity. The preferential transition, therefore, from the D^0,X initial state will be to the $2S$ final state. By similar arguments it can be shown that the initial state of the exciton bound to the first rotational state of the neutral donor will have even parity. The preferential transition for this state then will be to the $2P$ final state having odd parity. The intensities of these transitions were observed by Dean et al.[22] for the case of ZnTe. They showed that when the exciton collapsed from the first rotational state the terminal state intensity ratio $2P/2S=20$. They further showed that when the exciton collapsed from the D^0,X state the terminal state intensity $2P/2S=\frac{1}{5}$. We therefore conclude that in the case of GaAs the $2P$ final states are predominately associated with excitons bound to the first non-rigid-rotational state, and the $2S$ final states are predominately associated with the D^0,X state. The above techniques for identifying shallow donors, resulting from different chemical species, by photoluminescence are all associated with excitons bound to neutral donors. These same donors can also be identified, under appropriate conditions, from excitons bound to these donors in their ionized state (D^+,X). This technique has been applied in CdS (Refs. 17, 23, and 24) and ZnSe.[13] More recently, residual donors have been identifed in GaAs (Ref. 25) from the decay of excitons bound to the donor in the ionized state. In this paper we report the first identification of residual donors from the D^+,X transition in GaAs in the presence of a magnetic field. In many samples it is difficult to resolve D^+,X transitions in zero magnetic field

since they occur in the same spectral region as the free-hole—to—bound-donor transitions (D^0,h). The D^0,h transitions are broad relative to the D^+,X transitions and in many samples are of comparable intensity. In the presence of a magnetic field the D^+,X complex splits and the magnetic-field—split components are much more easily detected. The lines also sharpen due to the compressed wave function. We show the resolved structure of different donors using the photoluminescence technique. The donors are resolved from both the D^0,X and the D^+,X states in the presence of an external magnetic field. The magnetic field separation of the donors in the D^0,X state follows closely the expression derived by Fetterman et al.[26] The donors in the D^+,X state behave quite differently as would be expected. An exchange energy of approximately 30 μV was observed for the D^+,X state.

The identification of residual donors by photoluminescence has certain advantages over FTIR: (1) It can look through layers such as active layers as well as layers associated with some heterostructures, (2) it does not require contacts, (3) donors and acceptors can be identified simultaneously permitting an estimate of sample compensation, and (4) since it samples a very thin layer of material it can be used in profiling layers. An additional feature may be added to the photoluminescence technique described here which was introduced by Dean et al.[22] in ZnTe. The technique is called selective excitation of the donor—bound-exciton luminescence. The excitation is accomplished with a suitably narrow line of a tunable dye laser selected to excite the D^0,X state or one of its non-rigid-rotational donor states. Bound excitons can be created directly at these selected centers. If the time required for interdonor migration of the excitation by nonresonant tunneling transfer is long compared to the luminescence decay time of the center, then the inhomogeneous line broadening should be removed. This technique permits the identification of residual donors in material where the quality is somewhat degraded. It has been employed in ZnSe,[27] CdSe,[28] and InP.[29]

II. EXPERIMENTAL DETAILS

The samples used in this study were undoped, high-purity epitaxial layers grown on semi-insulating GaAs:Cr substrates by means of the H_2:AsCl$_3$:Ga vapor-deposition technique. Hall and electrical conductivity measurements were used to characterize the electrical parameters of the samples. Electron mobilities of the samples were typically greater than 10^5 cm^2/V sec at 77 K and their total concentration of electrically active impurities was $\sim 10^{14}$ cm^{-3}. For photoluminescence measurements the samples were mounted in a strain-free manner on one end of a sample holder which was, in turn, immersed in the tip of a glass helium Dewar containing a superfluid liquid-He bath whose temperature was maintained below 2.1 K. The Dewar tip was inserted in the air gap of a conventional dc electromagnet, the pole tips of which were separated by $\frac{5}{16}$ in. The maximum field strength of this magnet was 45 000 G. A krypton-ion laser radiating some 200 mW of cw power at 6471 Å was employed to pump the luminescence; spectral analysis of the photoluminescence was
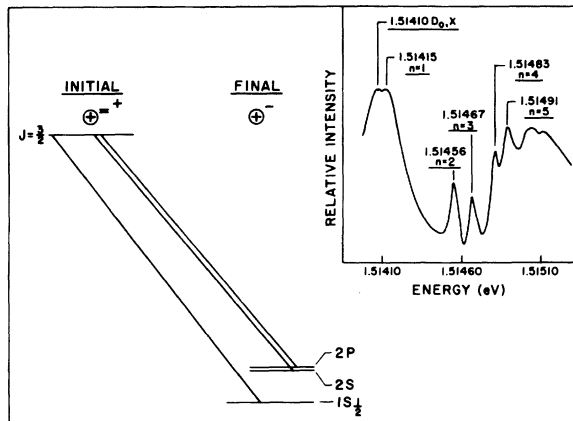
FIG. 1. Schematic representation of radiative recombination of an exciton bound to a neutral donor where the final state is the donor in the ground or in the excited configuration. The inset shows the initial state of the neutral-donor—bound exciton in the ground and several excited rotational states.
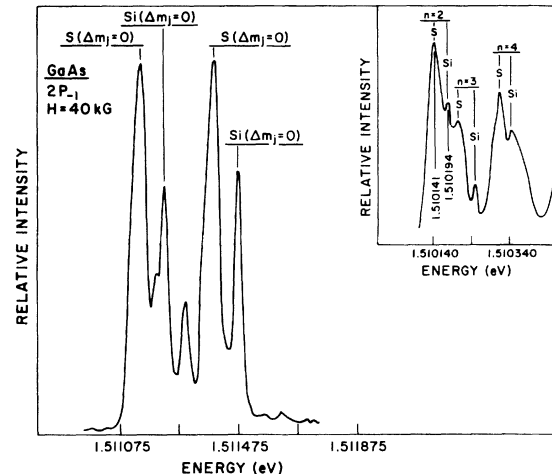

FIG. 2. Magnetic field components of the $2P_{-1}$ state at 40 kG resulting from the collapse of an exciton bound to the first-rotational state of the neutral donor for both sulfur and silicon. The inset shows the same two donors in zero magnetic field resulting from the exciton bound to the second, third, and fourth rotational states of these neutral donors.

achieved with a modified Bausch & Lomb 4-m grating spectrograph, equipped with a large (10-cm square) high-resolution diffraction grating ruled to 2160 grooves mm^{-1} and blazed at 5000 Å in first order. This instrument was capable of producing a first-order reciprocal dispersion of approximately 0.54 Å mm^{-1} over the wavelength range of interest. The photoluminescence spectra were photographically recorded on Kodak-type 1N spectroscopic plates. Wavelength calibration of the plates was achieved by nonlinearly interpolating the luminescence spectral lines, with respect to well-known interferometrically measured neon spectral lines, using the grating equation, the known geometry of the instrument, and the dispersion of the grating.

III. RESULTS AND DISCUSSION

A. Neutral donors

When an exciton decays from the D^0,X state in a magnetic field the excited $2S$ and $2P$ terminal states are separated. The $2P$ angular momentum states are also separated. The initial state of the complex consists of two paired electrons and one unpaired hole as shown in Fig. 1. The unpaired hole will split in a magnetic field. The final state of the complex consists of one unpaired electron either in the ground state or in an excited state. The transitions of interest in this paper are those associated with the terminal electron in the $n = 2$ state. The inset in Fig. 1 is a densitometer trace of the D^0,X state and its associated non-rigid-rotational states. The initial state of the transition can originate from D^0,X or any of its rotational states. In this paper we will be concerned with the transition whose initial state is the first non-rigid-rotational state. The specific transition results from the collapse of an exciton bound to the first non-rigid-rotational state and terminating in the $2P$ state of the electron on the neutral donor. It was shown by Dean et al.[22] in ZnTe that this transition terminating in the $2P$ state is 20 times more intense than the same transition terminating in the $2S$ state.

They also showed that the principle-donor—bound-exciton transition terminating in the $2S$ state is five times more intense than the same transition terminating in the $2P$ state. Since we only see a single transition to the $2P$ state we believe it to be the transition originating from the first non-rigid-rotational state. The initial state of the complex splits into a quartet and the final $2P$ state splits into three widely separated states, $2P_{+1}$, $2P_0$, and $2P_{-1}$. In this transition a negative diamagnetic shift occurs so that the lowest-energy $2P$ state results in the highest-energy optical transition. This is the $2P_{-1}$ state which has the highest intensity of the P-state transitions. The magnetic field splitting of this state at 40 kG is shown in Fig. 2. The inset in Fig. 2 shows the zero-field trace in the $n = 2$ spectral region of the neutral-donor—bound exciton. Here it is seen from the collapse of the exciton bound to the second, third, and fourth non-rigid-rotational states that the crystal contains residual Si and S donors. The splitting of the $2P_{-1}$ state shows components of these two donors as marked in Fig. 2. The highest-intensity transitions are the spin-conserving transitions. The lines are sharp and well resolved. The donors could not be resolved from this transition in zero magnetic field due to broadened lines and the near proximity of the principal-donor—bound exciton. Much can be gained by using a magnetic field with the photoluminescence identification of shallow donors.

It was shown by Fetterman et al.[26] that the separation between different chemical donors increased monotonically with magnetic field strength. In their case they were analyzing the donors by the FTIR technique. Since the exciton is very loosely bound to the donor in the photoluminescence scheme, a similar response might be expected. The perturbation theory of Fetterman et al. resulted in the following expression for the magnetic field separation of donors 1 and 2:

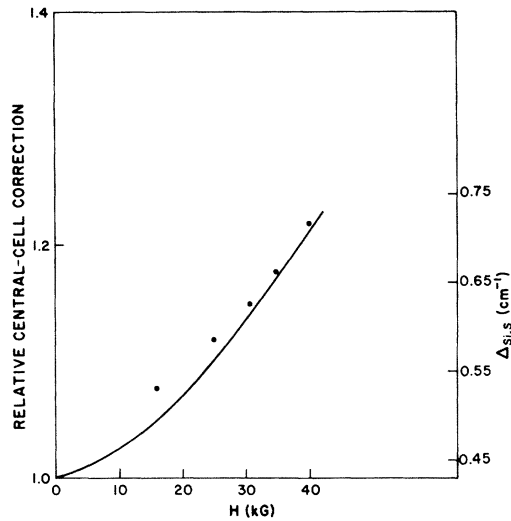$$\Delta_{1,2}(B) = K_{1,2} |X_{1s}(0)|^2 , \qquad (1)$$

FIG. 3. Separation of the Si and S donors as a function of magnetic field is shown by the closed circles. The solid line is the theoretical curve of $|X_{1S}(0)|^2$ as a function of magnetic field. The zero-field value of $\Delta_{Si,S}$ is 0.43 cm^{-1}.

where $K_{1,2}$ is an adjustable parameter independent of magnetic field. $X_{1s}(0)$ is the value of the effective-mass envelope function for an electron in the donor ground state at the origin in the presence of a magnetic field. This function is magnetic field dependent. X_{1s} can be calculated from effective-mass theory. The solid line in Fig. 3 is a theoretical plot of $|X_{1s}(0)|^2$ as a function of magnetic field taken from Cabib et al.[30] In the same figure we have plotted $\Delta_{Si,S}$ (silicon, sulfur donor separation) by adjusting the right-hand scale to place the point at 40 kG close to the theoretical curve. With the use of unit K, the remaining experimental points fall as shown in Fig. 3. The fit is reasonably good and shows that the perturbation theory is also applicable when an exciton is loosely bound to the donor. It is evident that at higher magnetic fields in-
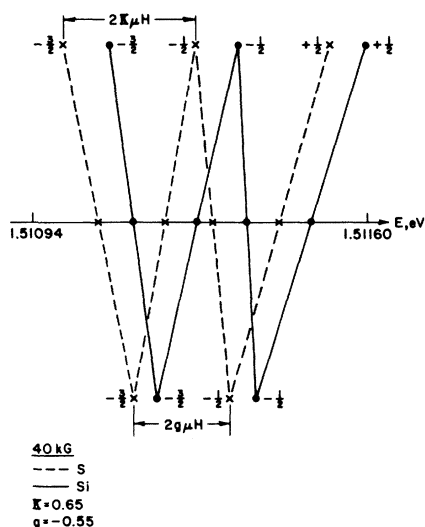

FIG. 4. Nomogram for interpretation of the $2P_{-1}$ magnetic field components of the Si and S donors. The free-electron g value is assumed, a hole κ value of $\kappa = 0.65$ is then measured.
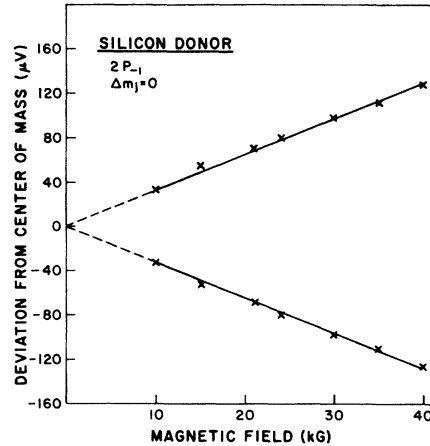

FIG. 5. Deviation from the center of mass is plotted as a function of magnetic field for the Si donor.

creased donor separation will occur.

We have used a geometric construction to analyze the magnetic field splitting of the $2P_{-1}$ states of the Si and S donors whose densitometer traces are shown in Fig. 2. Since the excitons are very loosely bound to the donors it is reasonable to assume that the electron g value in these bound states will be essentially the same as the free-electron g value. Using an electron g value of $g_e = -0.55$, we produced the nomogram at 40 kG shown in Fig. 4. From the nomogram a hole κ value of 0.65 is measured.

In Fig. 5 we have plotted the deviation from the center of mass of the $M_{J=0}$ transition for the silicon donor as a function of magnetic field. An effective g value of 1.1 is obtained from this splitting. This is close to the sum of the magnitudes of the electron and hole g values which is expected for this splitting. An identical splitting is obtained for the sulfur donor.

B. Ionized donors

Donors from different chemical species have been identified by photoluminescence spectra resulting from the collapse of excitons bound to these donors in the ionized state in CdS (Refs. 17, 23, and 24) and ZnSe.[13] In materials where the donor binding energy is smaller the identification of different donors becomes more difficult. It requires higher-quality material as well as higher-resolution techniques to successfully identify the residual donors in these materials. The identification of residual donors from excitons bound to the ionized state of these donors has recently been achieved in GaAs.[25] These measurements were made in zero magnetic field. Similar measurements have now been made, but in the presence of a magnetic field. A densitometer trace of the 40-kG magnetic field splitting of the Si and S D^+,X transition in GaAs is shown in Fig. 6. This is the same crystal in which the magnetic field splitting of the same donors in their D^0,X state is shown in Fig. 2. The identification of these donors in this crystal could not be made in zero magnetic field as they were masked by the D^0,h transition. In the inset in Fig. 6 the D^0,h transition is marked by the arrow; no evidence of the D^+,X transitions is seen. Here it is also ob-
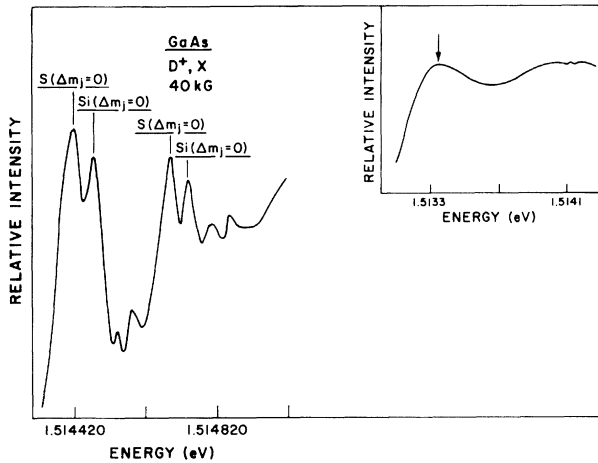
FIG. 6. Magnetic field components of the Si and S donors at 40 kG resulting from the collapse of an exciton bound to these donors in their ionized state. In the inset the arrow marks the position of the free-hole—to—bound-electron transition which masks the ionized-donor—bound-exciton transition.

FIG. 8. Plot of the magnitude of the magnetic field splitting of the π lines of the sulfur donor as a function of magnetic field.

served, as was the case for the D^0,X transitions, that the spin-conserving transitions have the highest intensity. The separation between the S and Si donors at 40 kG is 61 μV, which is only slightly larger than the separation of 53 μV at zero magnetic field.[4] The expression in Eq. (1) which describes the separation of donors as a function of magnetic field is not applicable to D^+,X transitions. In this expression the effective-mass envelope wave function is for the electron on the donor in the ground state. The donor electron is missing in the D^+,X complex. However, it might be expected that the electron from the exciton

will spend some time on the donor. Higher magnetic fields will be required to determine the effect of the magnetic field on the separation of different D^+,X states.

The magnetic field splitting of the π lines for the S donor is shown in Fig. 7. A solid curve is drawn through the experimental points. The dashed curve is a theoretical fit to the data.

It is clearly very difficult to calculate the behavior of the various radiative transitions associated with an exciton bound to an ionized donor in the presence of a magnetic field. This is mostly due to the fact that the hole is associated with the top valence band which is fourfold degenerate and highly anisotropic. Therefore, the behavior must be described by the Kohn-Luttinger Hamiltonian.[31] The Hamiltonian describing the D^+,X complex in a magnetic field is fairly complicated. However, one can obtain some idea about the variation of the energies of the π lines as a function of the magnetic field by assuming that the D^+,X complex behaves very similarly to the free exciton. This is not entirely unreasonable as the binding energy of a free exciton to ionized donor is small in GaAs. Hence we replace, for the purpose of studying its energy levels in a magnetic field, the D^+,X complex by a free exciton whose effective Rydberg is the sum of the free-exciton energy and the binding energy of the free-exciton bound to an ionized donor. Recently, Bajaj and Aldrich[32] have calculated the energy levels of a free exciton in an arbitrary magnetic field taking into full account the degeneracy and the anisotropy of the valence band using a variational approach. Using this calculation and the above-mentioned value of the effective exciton Rydberg along with the appropriate valence-band parameters,[32] we have evaluated the energies of the π lines as a function of the applied magnetic field. This variation is displayed in Fig. 7 as a dashed curve. We find that the values we calculate are in fair agreement with the experimental data thus indicating that our approximation is reasonable. In Fig. 8 we have
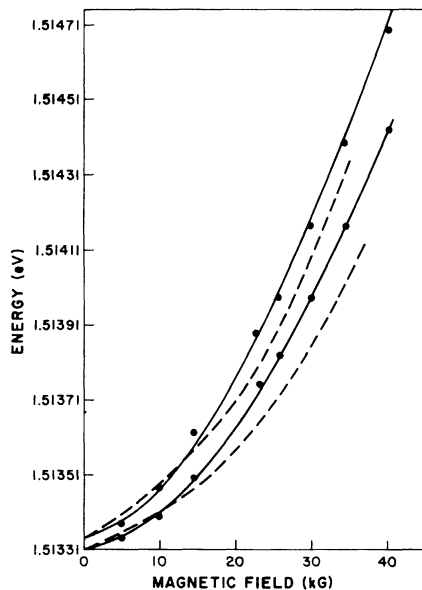

FIG. 7. Plot of the magnetic field splitting of the π lines of the sulfur donor. The transitions result from the collapse of an exciton bound to the donor in its ionized state. The closed circles are experimental points through which smooth curves have been drawn. The dashed curves represent an approximate theoretical prediction of the magnetic field splitting.
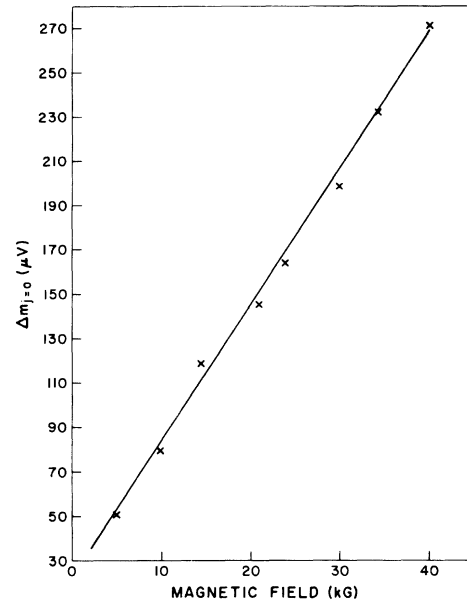
plotted the magnitude of the magnetic field splitting of the π lines for the S donor as a function of magnetic field. Extrapolating to zero magnetic field one obtains an exchange splitting of $\sim 30 \mu V$. Identical results were obtained for the Si donor.

IV. CONCLUSIONS

Residual donors were resolved in GaAs using photoluminescence techniques involving (D^0,X)-type complexes. The measurements were made in magnetic fields which sharpened the optical transitions and also separated the residual neutral donors. It made possible the identification of different chemical donors from transitions involving excitons bound to the first non-rigid-rotational state. These transitions are at least an order of magnitude more intense than transitions whose initial state consists of an

exciton bound to the second rotational state. This will permit the identification of residual donors at lower concentrations than would be possible by the previous technique.[5,6] The technique will also permit the identification of residual donors at higher concentration due to the line narrowing and donor separation. This will make it more convenient to compare with FTIR data at the same magnetic field.

Residual donors were also resolved from D^+,X complexes in the presence of a magnetic field. In many crystals this could not be done in zero field because the transition from a free hole to the bound donor comes in the same spectral region. This latter transition is much broader than the D^+,X transition and therefore masks it. The D^+,X components split in a magnetic field and are readily distinguishable from the D^0,h transition. The separation of different chemical donors from the D^+,X transitions in fields up to 40 kG is not appreciable.

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