Electronic properties of a (Cu-Li)-related neutral complex defect with a bound exciton at 2.25 eV in GaP

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GaP diffused with Cu and Li contains a large number of neutral ("isoelectronic") complexes, of which this paper is focused on one with a lowest electronic bound exciton (BE) line at ≈ 2.248 eV at 2 K. This lowest BE line is a spin triplet ($S=1$), which has a quite strong phonon coupling, so that the electronic line is not clearly observed. The true spectral dependence of the triplet photoluminescence (PL) emission could only be obtained via optical detection of magnetic resonance (ODMR). A singlet ($S=0$) transition with an electronic line at 2.2508 eV is observed in excitation spectra of ODMR or of PL. The angular dependence of ODMR data gives g values for the BE as $g_x=g_y=g_z=2.02\pm0.01$, i.e., quite isotropic, and D values $D_x=(-1.03\pm0.03)\times10^{-6}$ eV, $D_y=(-0.23\pm0.03)\times10^{-6}$ eV, and $D_z=(1.26\pm0.03)\times10^{-6}$ eV, where the axes x, y, and z diagonalizing the D tensor are defined as follows: **x** is 15° off $[\bar{1}\bar{1}2]$ towards [001], **y**$\|[1\bar{1}0]$, and **z** is 15° off [111] towards [110] in the $(1\bar{1}0)$ plane. The small values of the D components are due to unusually weak contributions from spin-orbit coupling and electron-hole magnetic dipole-dipole interaction. The symmetry of the defect is C_{1h}, and it is suggested to be a bent configuration of three atoms (Cu and Li).

I. INTRODUCTION

Photoluminescence (PL) spectroscopy for bound excitons (BE's) has been demonstrated to be a powerful technique to investigate the electronic structure of defects in semiconductors,[1] in particular if combined with selective excitation with tunable dye lasers[2–4] and with optical detection of magnetic resonance (ODMR).[5] In this work a complex neutral ("isoelectronic") Cu- and Li-related defect in GaP is studied with these techniques. Previous studies on related defects in GaP (Refs. 4 and 6–15) have shown that the electronic structure of the corresponding BE's is typically triplet-singlet (TS) pairs, i.e., the lowest-energy BE states are built up from an electron-hole pair where both particles have spinlike character.[8,16,17] Rather large variation occurs within this general electronic structure, when it comes to details such as phonon coupling, singlet-triplet (S-T) splitting energies [i.e., electron-hole (e-h) exchange splitting], and ODMR spectra.[6–15] In general the electronic structure of these defects is now rather well understood,[8,18,19] but their identification is still speculative.

ODMR is particularly suitable for the study of such neutral defects which give rise to a spin triplet as the lowest BE state.[4,6,11–15,20–24] Due to the large linewidth of ODMR resonances in GaP, hyperfine structure is only occasionally resolved.[22,25–29] Nevertheless, valuable symmetry information is usually obtained, leading to a specific geometric model for a complex defect.[14,15,20,21] Selectivity is important in ODMR, since excitation transfer between defects may cause detection of ODMR signals via one defect spectrum, while the resonance actually occurs in another defect.[26,30,31] The combination with selective dye-laser excitation may here be very helpful.[5]

The (Cu-Li)-related defect studied in this work is in our opinion an excellent demonstration of the power of this combination of two different spectroscopies. The PL spectrum of the triplet BE state can only be measured properly with detection in phase with the microwave resonance, and it is drastically enhanced by selective excitation. The singlet BE state can only be seen in PL at elevated temperatures, or by scanning the dye-laser excitation photon energy, while detecting in the triplet PL spectrum (with or without microwave resonance). Excitation transfer effects between defects are directly demonstrated in this work.

II. SAMPLES AND EXPERIMENTAL PROCEDURE

The samples used in this study were prepared by a two-step diffusion procedure, first diffusing Cu at $\approx 900\,^{\circ}$C and subsequently Li at lower temperatures. The conditions of doping were the same as previously described for samples showing the $(\text{Cu-Li})_V$ defect with a BE at 2.172 eV.[13] The complex defect studied in this work was only seen in such (Cu-Li) co-doped material and was never seen in GaP which was diffused with Cu only, Li only, or anything else. We therefore conclude that the defect contains both Cu and Li atoms.

Photoluminescence spectra were recorded in an optical setup, containing a CryoVac He cryostat with continuous variation of temperature, and a Jobin Yvon HRD1 0.6-m double monochromator. Excitation was obtained either from an Ar^+ laser or a Coherent 590 dye laser, with a Coumarin 540 dye. ODMR spectra were obtained in both the Faraday and the Voigt configuration, at temperature variable down to 2 K in a modified 9-GHz Bruker ESR 200D-SRC spectrometer, with a cylindrical cavity. A 0.25-m Jobin Yvon grating monochromator was also used in the ODMR setup, for spectral studies.

III. EXPERIMENTAL RESULTS

A. Optical spectra for the 2.25-eV bound exciton

A typical PL spectrum at 2 K of a (Cu-Li) co-doped crystal with 5145-Å Ar^+-laser excitation (above the band gap of GaP) is shown in Fig. 1(a). The visible part of the spectrum is dominated by two different (Cu-Li)-related spectra, one peaking at ≈ 2.20 eV, the so-called $(Cu\text{-}Li)_{III}$ spectrum,[12] and another peaking at 2.13 eV, with a sharp no-phonon line at 2.172 eV, i.e., the $(Cu\text{-}Li)_V$ spectrum.[13] Spectra related to N_P (Ref. 32) and $Li_i\text{-}Li_{Ga}\text{-}O_P$ (Ref. 33) are also observed. These spectra are all unrelated to the defect studied in this work. The 2.25-eV BE particularly discussed in this work has a broad PL band peaking at about 2.14 eV, which is hidden in the background of the spectrum in Fig. 1(a).

At somewhat elevated temperatures the 2.25-eV BE can be observed in PL emission, since the electronic singlet line at 2.2508 eV is then thermally populated

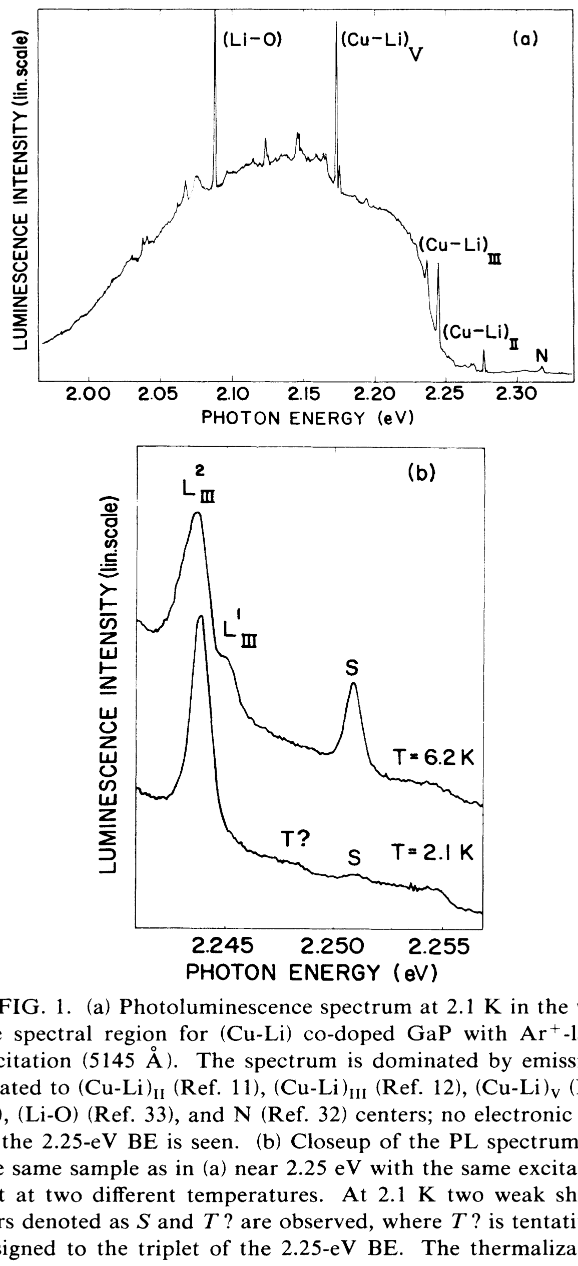

FIG. 1. (a) Photoluminescence spectrum at 2.1 K in the visible spectral region for (Cu-Li) co-doped GaP with Ar^+-laser excitation (5145 Å). The spectrum is dominated by emissions related to $(Cu\text{-}Li)_{II}$ (Ref. 11), $(Cu\text{-}Li)_{III}$ (Ref. 12), $(Cu\text{-}Li)_V$ (Ref. 13), (Li-O) (Ref. 33), and N (Ref. 32) centers; no electronic line of the 2.25-eV BE is seen. (b) Closeup of the PL spectrum for the same sample as in (a) near 2.25 eV with the same excitation but at two different temperatures. At 2.1 K two weak shoulders denoted as *S* and *T*? are observed, where *T*? is tentatively assigned to the triplet of the 2.25-eV BE. The thermalization between lines *S* and *T*? is clearly seen at a slightly elevated temperature 6.2 K, with the singlet *S* at 2.2508 eV dominating over the triplet *T*? due to its much stronger oscillator strength. Lines denoted by L^1_{III} and L^2_{III} belong to another center, $(Cu\text{-}Li)_{III}$ (Ref. 12).

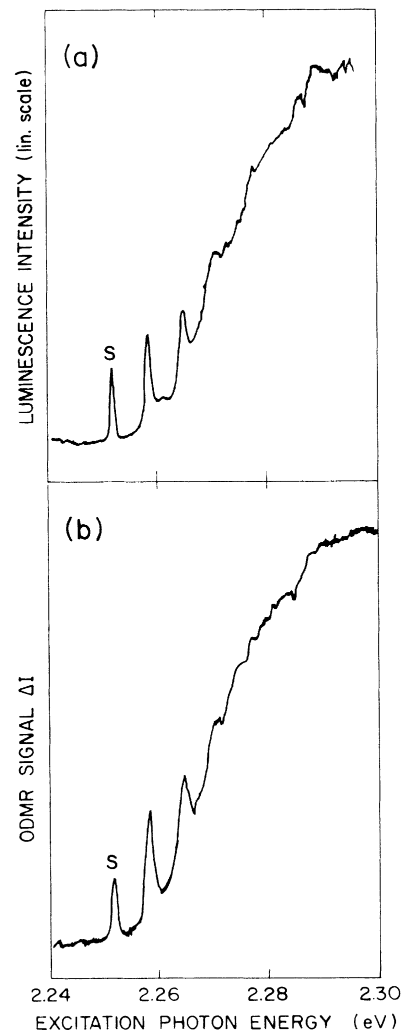

FIG. 2. (a) Photoluminescence excitation spectrum of the 2.25-eV BE obtained at 2 K using a Coherent 590 dye laser with a Coumarin 540 dye, with detection at about 2.20 eV. The singlet BE line denoted *S* is seen clearly, with phonon replica of a quasilocalized phonon mode of about 7 meV towards higher energies. (b) Similar excitation spectrum as in (a), but with ODMR detection in the resonance at $B \approx 0.33$ T, shown below in Fig. 6. This ODMR-PLE spectrum is a fingerprint of the 2.25-eV BE, proving that the 2.25-eV BE transition is detected in ODMR.

[Fig. 1(b)]. A broad and weak shoulder at 2.248 eV at 2.1 K [Fig. 1(b)] is believed to be the triplet line of this BE.

The existence of the 2.25-eV BE is most easily demonstrated at 2 K in photoluminescence excitation (PLE) spectra, as shown in Fig. 2(a). Here the detection is set at ≈ 2.20 eV, and the dye laser is scanned over the photon energies shown. A sharp line is observed at ≈ 2.2508 eV, with phonon replica of a quasilocalized phonon mode of about 7 meV towards higher energies. A broad phonon-assisted background is also seen towards higher energies. This line at 2.2508 eV is identical to the line observed at the same energy in Fig. 1(b), at 6.2 K. A similar PLE spectrum is observed under conditions of magnetic resonance, in a part of the ODMR resonance spectrum centered around $B=0.33$ T, as shown in Fig. 2(b). The details of the ODMR spectrum are discussed further in Sec. III B below.

The PLE spectrum in Fig. 2(a) was investigated with the sample placed in a magnetic field of 6 T in the Voigt configuration. As shown in Fig. 3, no splitting of the 2.25-eV line was seen at this field, neither at 2 K nor at elevated temperatures. Consequently it can be firmly concluded that this strong line is derived from a transition between two magnetic singlets, i.e., between an $S=0$ BE state and an $S=0$ ground state.

As noted above no PL lines corresponding to the electronic transitions of the 2.25-eV BE are convincingly observed at 2 K, only two weak shoulders can be seen, at ≈ 2.248 eV and ≈ 2.251 eV, respectively [Fig. 1(b)]. This is a proof that the singlet state at 2.2508 eV is not the lowest electronic state of the BE, as expected, since the triplet state is normally observed at lower energy. The triplet state usually has much lower oscillator strength than the singlet state, consequently it is not observed in PLE in Figs. 2 and 3.

To obtain a clean PL spectrum for the triplet line corresponding to the 2.25-eV BE, a selective excitation within the range of the absorption spectrum of the corresponding singlet line is necessary, assuming that the relaxation time from the singlet state down to the triplet is rather short. This procedure is not sufficient to selectively excite the triplet spectrum, however, since other defect BE's, such as the 2.172-eV $(\text{Cu-Li})_V$ defect, also have absorption bands in the same spectral region ($\approx 2.25-2.32$ eV). The only way to get a selective spectrum for the triplet is to detect the spectral dependence of the triplet ODMR signal. This was successfully performed in this work, as shown by curve (c) of Fig. 4, where also the ordinary PL spectrum is shown for comparison [curve (a) of Fig. 4]. The recording in trace (c) of Fig. 4 was done with $\mathbf{B}\|[111]$ and $B\approx 0.33$ T, under which conditions no ODMR resonance occurs from $(\text{Cu-Li})_V$ centers. Consequently the overlapping from $(\text{Cu-Li})_V$ centers is avoided and possible transfer effects are not influencing the spectrum (see also below Sec. III C). The corresponding selective spectrum for the $(\text{Cu-Li})_V$ triplet BE state is also shown in trace (b) of Fig. 4. The observed 2.25 eV triplet BE spectrum is quite broad and featureless, with a full width at half maximum (FWHM) of about 0.12 eV. The electronic triplet line is quite weak and cannot be resolved. The absence of structure in the broad phonon envelope shows that a broad range of phonons couple to the electronic triplet transition of this defect. This is probably the reason why the electronic triplet line is not observed even at 2 K.[34,35] The exact position of the electronic

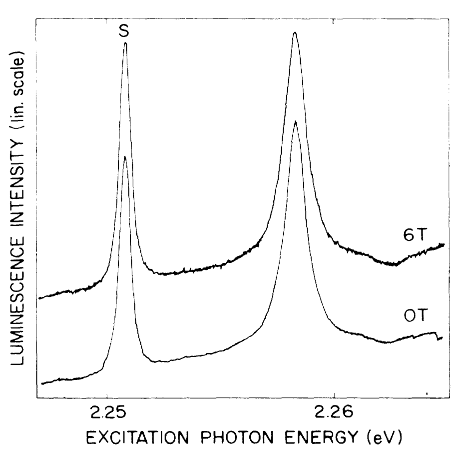

FIG. 3. PLE spectra at 2 K of the 2.25-eV BE similar to that shown in Fig. 2(a), compared with the case of an applied magnetic field of 6 T in the Voigt configuration. No sign of splitting for the singlet S is seen, neither at 2 K (in the figure) nor at elevated temperatures (not shown).

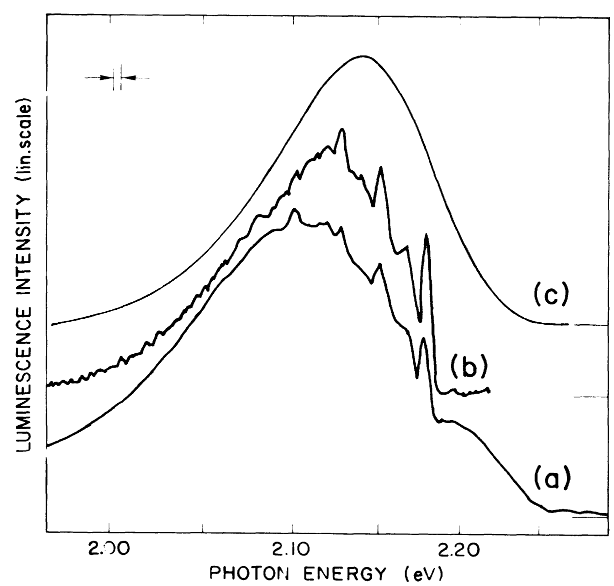

FIG. 4. Curve (a) shows a PL spectrum of the same sample as in Fig. 1, but at 4 K and with lower spectral resolution, as a relevant comparison with the spectral dependences of ODMR in the same experimental conditions. Curve (b) is the spectral dependence of ODMR from the $(\text{Cu-Li})_V$ center with $B\approx 0.13$ T [see Fig. 5(a) below]. Curve (c) shows the ODMR spectral dependence of the 2.25-eV center with $B\approx 0.33$ T, which is the true triplet PL spectrum of the 2.25-eV center, but which is buried in the background of other PL emissions if an ordinary PL spectrum is taken, as seen in curve (a).

triplet line cannot be determined, from Fig. 4 it appears likely that it is close to 2.25 eV, in agreement with the PL data in Fig. 1(b), where the triplet seems to be about 3 meV below the 2.2508 eV singlet line, i.e., at 2.248 eV.

B. ODMR spectra

The ODMR spectrum of the 2.25-eV BE was measured in photoluminescence, i.e., the excited (BE) state was investigated. It was essential to carefully select optimal experimental conditions in order to get clean ODMR data for this BE. In Fig. 5(a) is shown an ODMR spectrum at 4 K with above-band-gap laser excitation, displaying resonances from the $(Cu\text{-}Li)_V$ BE (Refs. 13 and 21) as well as from the 2.25-eV BE (the two lines at $B \approx 0.33$ T). The detection photon energy was in this case close to the peak position of the 2.25 eV triplet, i.e., close to 2.15 eV. To isolate the ODMR resonances associated with the 2.25-eV BE, selective excitation below the band gap (but above 2.25 eV) could be used, as shown in Fig. 5(b), where the $(Cu\text{-}Li)_V$ ODMR resonances have nearly disappeared. They are completely absent if the detection photon energy is set, e.g., at 2.20 eV, as in Fig. 5(c), since this energy is above the $(Cu\text{-}Li)_V$ BE electronic lines.[13] A raise in temperature to about 10 K brings down the $(Cu\text{-}Li)_V$ ODMR resonance [Fig. 5(d)], since in this situation the triplet line in

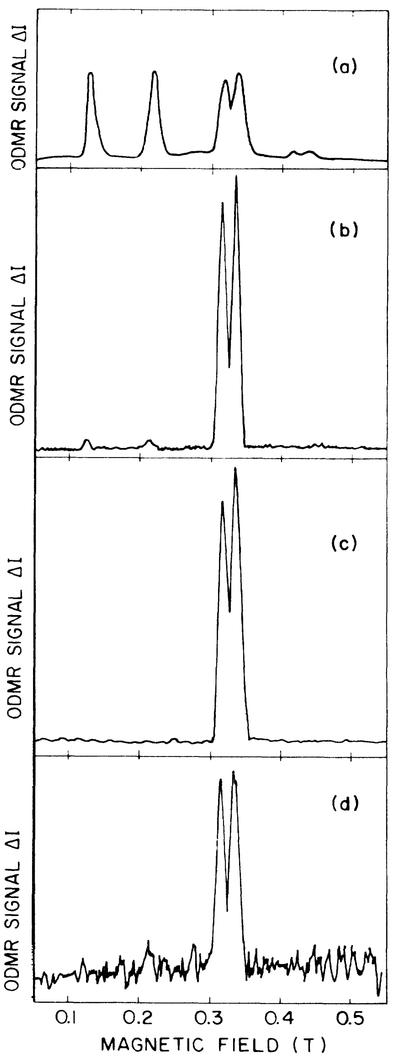

FIG. 5. ODMR spectra with $\mathbf{B}\|[111]$, (a) at 4 K with above-band-gap excitation (5145 Å) and with detection of the whole visible PL emission, displaying ODMR from the $(Cu\text{-}Li)_V$ BE as well as the 2.25-eV BE (the middle field resonances); (b) at 4 K with a selective excitation below the band gap (but above 2.25 eV) and a detection of the whole PL band below 2.25 eV, showing a nearly complete disappearance of the $(Cu\text{-}Li)_V$ ODMR resonance; (c) at 4 K with below-band-gap excitation and with detection at about 2.20 eV, showing only the ODMR from the 2.25-eV BE; (d) with the same excitation and detection as in (a) but at a somewhat higher temperature, about 10 K, showing a nearly complete quenching of the $(Cu\text{-}Li)_V$ ODMR signals.

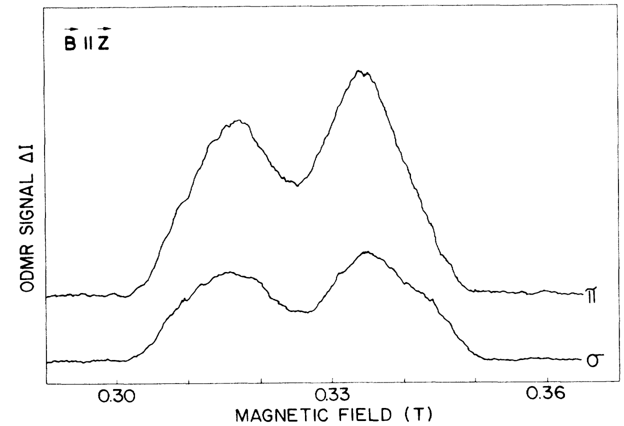

FIG. 6. ODMR spectra obtained for the 2.25-eV BE at 4 K at a microwave frequency of 9.22 GHz with **B** along the principal axis z. The Voigt configuration was employed, and spectra obtained in both π and σ polarizations are shown.

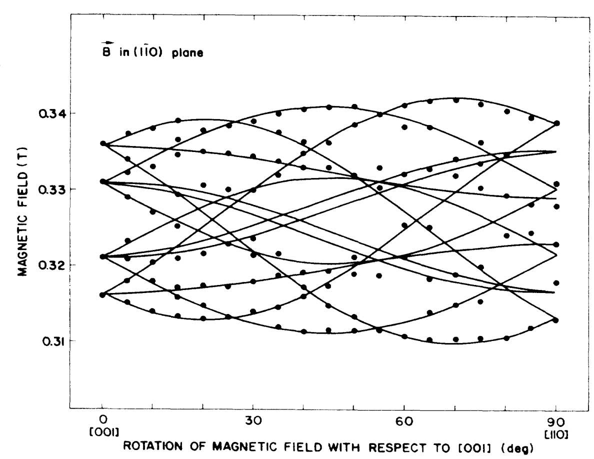

FIG. 7. Angular dependence of ODMR spectra for the 2.25-eV BE, for rotation of the sample with **B** in the $(1\bar{1}0)$ plane. Dots show experimental data, while the solid lines are obtained from the fitting of the spin Hamiltonian given in the text, with the parameters in Table I.

the (Cu-Li)$_V$ BE is very weak, and the (Cu-Li)$_V$ emission is dominated by the singlet, with the electronic line at 2.174 eV.[13] Above 10 K the ODMR signal from the 2.25 eV triplet also gradually quenches, but much less than the (Cu-Li)$_V$ ODMR signal.

Using proper conditions, as in Fig. 5(c), the ODMR resonance associated with the 2.25-eV BE was investigated in detail. The spectrum consists of a group of lines overlapping in a narrow region of field which makes detection of different polarization components necessary, as evident from Fig. 6, where both σ and π polarizations are shown. The angular dependence of this spectrum was carefully studied in the Voigt configuration with the magnetic field **B** rotated in the $(1\bar{1}0)$ plane (Fig. 7). A rather complex splitting pattern is obtained, it is, e.g., found in Fig. 7 that the maximum splitting occurs for **B** between [111] and [110], 15° off [111], in the $(1\bar{1}0)$ plane. Obviously the defect is of low C_{1h} symmetry. The ODMR splitting pattern is consistent with a pair of triplet resonances ($\Delta M = \pm 1$), where the $\Delta M = 2$ resonance sometimes observed for other defects[17,20,21,24] is completely absent.

A theoretical fit to the experimental data in Fig. 7 with a spin-triplet-spin Hamiltonian

$$\mathcal{H} = \mu_B \mathbf{B} \cdot \overleftrightarrow{\mathbf{g}}_{ex} \cdot \mathbf{S} + \mathbf{S} \cdot \overleftrightarrow{\mathbf{D}} \cdot \mathbf{S} ,$$

gives the parameters listed in Table I. In the fitting procedure a basis set of spinlike wave functions was used for both electron and hole states. The deviation of the g tensor g_{ex} from $g=2$ is very small indeed in this case, and no clear residual anisotropy can be resolved. The signs of the D components are experimentally determined from polarized data taken in the Faraday configuration. The components of the D tensor are quite small compared to other complex defects related to Cu and Li in GaP.[14,20,21]

C. Excitation transfer detected by ODMR

Excitation transfer effects may sometimes be important in ODMR,[5] and were carefully investigated in the present case. This is illustrated in Fig. 8, where a PLE spectrum is observed under resonance conditions in the (Cu-Li)$_V$ triplet, i.e., the resonant change in the $\Delta M = 2$ resonance at $B \approx 0.13$ T [Fig. 5(a)] is monitored. This PLE spectrum shows a weak excitation from the (Cu-Li)$_V$ defect, peaking at about 2.22 eV and another rise above 2.25 eV, due to the 2.25-eV BE. The phonon structure associated with the 2.25-eV BE [Fig. 2(a)] is actually seen, as shown in the inset. To find out whether ODMR in the 2.25-eV BE directly contributes to this PLE spectrum a PL spectrum is taken by monitoring the same $B \approx 0.13$ T resonance under conditions of excitation in the 2.25 eV defect absorption spectrum, as shown in Fig. 4(b), apparently a fingerprint of (Cu-Li)$_V$ PL. This is interpreted as evidence for excitation transfer from the 2.25 eV to the (Cu-Li)$_V$ defect, i.e., the BE is transferred between these two defect sites before recombining as a (Cu-Li)$_V$ BE. These effects of excitation transfer are avoided in this study of the 2.25-eV BE by careful selective detection, as described above. The conclusions on the connections between ODMR spectra and the respective (Cu-Li)-related defects discussed above therefore remain perfectly valid.

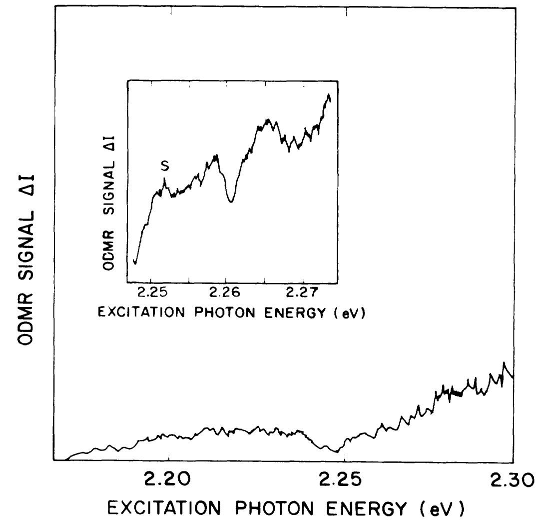

FIG. 8. ODMR-PLE spectrum for the (Cu-Li)$_V$ BE with detection in the resonance at $B \approx 0.13$ T as shown in Fig. 5(a). It shows, besides a (Cu-Li)$_V$-related phonon-assistant excitation spectrum, a comparably stronger excitation with no-phonon line at about 2.25 eV and a quasilocalized phonon mode of about 7 meV (seen weakly in the inset), which is the fingerprint of the 2.25-eV BE. This is interpreted as involving a weak excitation transfer process between the 2.25 eV and the (Cu-Li)$_V$ complex.

TABLE I. Spin Hamiltonian parameters for the 2.25 eV bound exciton, where **x** is 15° off $[\bar{1}\bar{1}2]$ towards [001] in the $(1\bar{1}0)$ plane; **y**$\|[1\bar{1}0]$; **z** is 15° off [111] towards in the (110) plane.

	Parameter	Value
$\overleftrightarrow{g}$ tensor	g_x	2.02 ± 0.01
	g_y	2.02 ± 0.01
	g_z	2.02 ± 0.01
$\overleftrightarrow{D}$ tensor	D_x	-1.03 ± 0.01
(10^{-6} eV)	D_y	-0.23 ± 0.01
	D_z	1.26 ± 0.01

IV. DISCUSSION AND CONCLUSIONS

The data displayed in this work demonstrate the power of a combination of ODMR and selective excitation in entangling the detailed electronic properties of a complex neutral (isoelectronic) defect, whose optical spectrum is completely buried in the background of other PL emissions, if conventional PL spectroscopy with above-band-gap laser excitation is employed. The defect studied here belongs to a rather common class in GaP doped with Cu and/or Li, namely low-symmetry neutral complexes, dominated by a hole-attractive potential, in this case Cu_{Ga}, a deep double acceptor on Ga site.[36] A strongly hole-attractive potential combined with a low

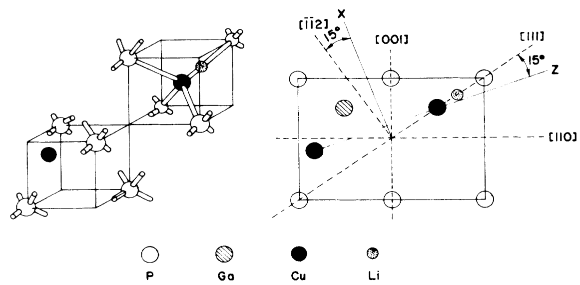

FIG. 9. A possible model of the suggested defect identity for the 2.25 eV complex as Cu_i-Cu_{Ga}-Li_i, where the Cu_i is relaxed away from the [111] crystalline axis by about 15° towards [110] in the $(1\bar{1}0)$ plane, i.e., sitting somewhere between the three tetrahedral interstitial sites. x, y, and z denote the principal axes of the defect, as described in the text.

symmetry is likely to cause quenching of the orbital angular momentum of the bound hole in GaP, and a triplet-singlet pair is expected at lowest energy for the BE.[8,16] For the 2.25-eV BE the e-h exchange splitting is determined to be about 3 meV, a value similar to BE's for other (Cu-Li)-related complexes in GaP with a similarly delocalized secondary electron in the BE.[11–13] The rather strong phonon coupling in this case is consistent with one of the particles (the hole) being strongly localized at the defect. The observed differences in phonon coupling selection rules between the singlet and triplet states are common for these types of BE's in GaP.[17]

The anisotropy δg_{ex} for the g_{ex} tensor for the BE was not resolved in this case, and thus smaller than $\delta g_{ex} = 0.5\%$, the smallest anisotropy observed so far for complex defects in GaP. For the case of the 1.911-eV Cu-related BE in GaP studied earlier, the anisotropy was small but still about 2.5%.[20] For all other BE's in the same spectral region, the residual anisotropy was found to be larger.[14,15,21] The g_{ex} tensor for the BE can be written $\overleftrightarrow{g}_{ex} \approx \frac{1}{2}(\overleftrightarrow{g}_e + \overleftrightarrow{g}_h)$ if the anisotropy is small and the values of both $\overleftrightarrow{g}_e$ and $\overleftrightarrow{g}_h$ are similar.[20] Here $\overleftrightarrow{g}_e$ and $\overleftrightarrow{g}_h$ are the individual g tensors for the bound electron and hole, respectively. $\overleftrightarrow{g}_e$ is usually found to be quite isotropic and very close to $g_e = 2.00$ in GaP, at least for shallow donorlike electrons.[32] The hole is expected to show some anisotropy, due to spin-orbit coupling,[18] apparently a very small effect in this particular case.

The D tensor is also about an order of magnitude smaller than for other comparable complexes in the same spectral energy range,[14,20,21] at least partly due to the very small spin-orbit effects for the bound hole. The magnetic dipole-dipole term must also be quite small in this case, however. A likely explanation might be that one of the bound carriers (the electron) is quite delocalized, while the other (the hole) is very localized.

The identity of the defect giving rise to the 2.25-eV BE partly remains a matter of speculation, since no hyperfine interaction could be resolved in the ODMR resonances, as was also the case in previous ODMR studies of similar defects in the same spectral range.[14,15,21] Cu_{Ga} is probably responsible for the hole-attractive part, two interstitials of Cu or Li are required in addition to create a neutral isoelectronic defect. The C_{1h} symmetry concluded from ODMR data suggests a bent configuration, i.e., all three atoms are not situated along the same axis. A possible example of such a configuration is given in Fig. 9. The model in Fig. 9 is drawn so that the principal axis z of the defect passes through the interstitial Cu_i and the center of the Cu_{Ga}-Li_i pair where the two interstitial atoms are arranged in an unsymmetric configuration at two different sides of Cu_{Ga}.

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