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Optical detection of conduction-electron spin resonance in GaAs, $Ga_{1-x}In_xAs$, and $Ga_{1-x}Al_xAs$

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The optical detection of conduction-electron spin resonance (CESR) is performed in GaAs, $Ga_{1-x}In_xAs$, and $Ga_{1-x}Al_xAs$ alloys. The measured g factor of GaAs is $g^* = -0.44 \pm 0.02$. The good precision obtained permits a fruitful comparison with theory. It is shown that such measurements are very sensitive to optically created hyperfine nuclear fields which may shift noticeably the CESR line. A simultaneous nuclear saturation is then mandatory to obtain significant results.

I. INTRODUCTION

The measurement of the Landé factor g^* of conduction electrons in semiconductors is of interest since it is one of the few quantities which can be easily calculated through $\vec{k}\cdot\vec{p}$ perturbation theory.[1] We show in the following paper[2] that, together with the effective-mass value, the knowledge of the g factor permits to obtain a precise determination of the momentum interband matrix element, which is a fundamental parameter of $\vec{k}\cdot\vec{p}$ theory. The most precise measurements of g factors are due to conduction-electron spin-resonance (CESR) experiments. Unfortunately, the number of semiconductors in which standard EPR techniques[3] can be applied to detect CESR is quite small: as a matter of fact the technique requires the incorporation of a fair amount of donor impurities which can lead to line broadening rending this technique unpractical. Up to recently, the only g factors of III-V compounds determined by resonance techniques were those of InSb,[4,5] InAs,[6] and GaAs.[7] As is shown in Ref. 2, the two compounds InSb and InAs are not the most interesting to test $\vec{k}\cdot\vec{p}$ theory approximations as, due to their small bandgaps, the three-band approximation of $\vec{k}\cdot\vec{p}$ theory is accurate. In other words, it means that Roth's formula is correct, that is,[8]

$$\frac{g^*}{g_0} = 1 - \frac{\Delta_0}{3E_0 + 2\Delta_0}\left(\frac{m_0}{m^*} - 1\right), \qquad (1)$$

where g^* is the effective g factor, m^* the effective mass, E_0 the band gap, Δ_0 the spin-orbit splitting of the valence band, g_0 and m_0 the g factor and mass of the free electron. It is clear that the knowledge of g^* is then equivalent to the knowledge of m^*. The early conventional EPR measurement[7] in n-type GaAs does not seem to have been fully exploited to test the approximations leading to (1). Anyway, this would have led to wrong conclusions, as in Ref. 7 a wrong positive sign is attributed to the g factor of GaAs. The sign of the g factor of GaAs was questioned up to recently, as for instance Vekua *et al.*[9] claim to also measure a positive sign. It is only three years ago that White *et al.*[10] rightly evidenced the negative sign of the g factor. The failure of Eq. (1), proving the inadequacy of the three-band approximation, was thus only recently acknowledged,[11] due to a new technique of CESR which can be applied in pure or p-type material: the optical detection of CESR,[12] which relies on the spin orientation of carriers by absorption of circularly polarized intrinsic light. This technique has already been applied to GaSb,[12] and InP,[13] and we report here its application to GaAs and the alloys $Ga_{1-x}In_xAs$ and $Ga_{1-x}Al_xAs$.

III-V alloys have been much less extensively studied than pure compounds. The practical parameters of importance for devices (room-temperature band gaps, radiative efficiencies,...) have been measured,[14] but more fundamental parameters were overlooked. Most effective-mass measurements were carried out at room temperature,[15] where their interpretation is delicate (see the discussion in Ref. 2). Only in $Ga_{1-x}In_xAs$ was the effective mass precisely measured at low temperatures by cyclotron resonance in high magnetic fields.[16] Our CESR measurements in $Ga_{1-x}In_xAs$ and $Ga_{1-x}Al_xAs$ alloys performed at low magnetic fields ($\lesssim$400 G) directly yield an unambiguous value of the g factor of conduction electrons at the bottom of the conduction band, thus permitting a precise comparison with calculated g factors. Another interesting feature is the observation of narrow resonance lines, which shows that alloy disorder does not induce band parameter broadening, as was already observed on optical and transport properties.

II. PRINCIPLES OF THE OPTICAL DETECTION OF CESR IN SEMICONDUCTORS

A. Optical spin orientation of conduction electrons

It is now well established that optical pumping in semiconductors by circularly polarized light leads to spin polarized conduction electrons.[17] The spin polarization and its variations are conveniently measured by detecting the degree of circular polarization of the recombination light. We give here the main results of the previous analyses, emphasizing the aspects relevant to CESR.

The electronic spin polarization P is defined by

$$P=(n_+-n_-)/(n_++n_-)\,,$$

where n_+ and n_- are the densities of electrons with up or down spin along the direction of propagation of the exciting light, z. For electrons created at the bottom of the conduction band in III-V cubic compounds excited with σ_+ circularly polarized light, the steady-state polarization in zero magnetic field is given by[18]

$$P=-0.5T_1/(T_1+\tau)\,, \qquad (2)$$

where τ is the lifetime and T_1 the spin-relaxation time of conduction electrons. When a longitudinal magnetic field B_z is applied, T_1 can be a function of the field $T_1(B_z)$ and thus P varies with the applied longitudinal field.[19] In zero magnetic field, the value $T_1(0)$ is equal to T_2, the relaxation time of the component of the electronic magnetization transverse to a magnetic field.

The degree of circular polarization of the recombination light observed along the z axis is defined by

$$\mathcal{P}=(L_+-L_-)/(L_++L_-)\,,$$

where L_+ and L_- are the intensities of the recombination light with σ_+ or σ_- circular polarization. In III-V compounds, $\mathcal{P}$ is related to P by[18]

$$\mathcal{P}=-0.5P\,.$$

As the CESR signal is detected on the polarization of the luminescence light, it is useful to observe a large polarization signal to have a good signal-to-noise ratio on the CESR signal. In this connection, it has been observed in several compounds (GaSb,[20] GaAs,[21] $\mathrm{Ga}_{1-x}\mathrm{In}_x\mathrm{As}$,[22] $\mathrm{Ga}_{1-x}\mathrm{Al}_x\mathrm{As}$,[23] and[22] InP) that the photocreation of carriers with an exciting energy significantly larger than the band gap can lead to small polarizations. This may be due to an increase of the spin-relaxation rate for hot carriers created slightly above the band edge. At still higher energies ($h\nu > E_0+\Delta_0$), the excitation from the split-off Γ_7 valence band produces electrons which have an opposite polarization with respect to those originating from the Γ_8 upper valence band. It is thus often important to excite the semiconductor with near-band-gap photons.

The application of a transverse magnetic field B_x produces a precession of the oriented conduction electrons and consequently depolarizes the recombination light.[18] The depolarization curve is Lorentzian with a half-height width given by

$$\Delta B=\frac{\hbar}{|g^*|\,\mu_B}\left(\frac{1}{T_2}+\frac{1}{\tau}\right), \qquad (3)$$

where g^* is the g factor of the conduction electrons. The combined measurements of $\mathcal{P}$ in zero applied field and of ΔB yield T_2 and τ, provided that g^* is known. On the other hand, a direct measurement of τ by a transient technique permits to calculate T_2 from the value of $\mathcal{P}$ in zero field. A measurement of ΔB then gives $|g^*|$. This nonresonant determination of g^* was recently used by Chadi *et al.* in $\mathrm{Ga}_{1-x}\mathrm{Al}_x\mathrm{As}$ alloys.[24]

B. Optical detection of CESR

Consider a sample excited with circularly polarized light in a longitudinal magnetic field B_z. If we irradiate it with a transverse rf field B_1 of frequency ω_0, transitions between the two spin levels occur if the resonance condition $\hbar\omega_0$ $\hbar\omega_0=|g^*|\mu_B B_0$ is fulfilled. The population difference between the spin levels is resonantly decreased, and this decrease will be reproduced on the circular polarization of the luminescence light allowing the optical detection of the resonance. The steady-state solution of the Bloch equations governing the evolution of P gives the relative decrease of the electronic polarization[12]

$$\frac{\Delta P}{P}=\frac{(T_{1*}/T_{2*})B_1^2}{(T_{1*}/T_{2*})B_1^2+(\Delta B)^2+(B_z-B_0)^2}\,, \qquad (4)$$

where $1/T_{1*}=1/T_1+1/\tau$ and $1/T_{2*}=1/T_2+1/\tau$.

From this equation, one sees that the decrease is appreciable if the saturation factor $s=(T_{1*}/T_{2*})(B_1/\Delta B)^2$ is of the order of unity, that is if $(g^*\mu_B/\hbar)^2T_{1*}T_{2*}B_1^2\simeq 1$. In our experiments, T_1 and T_2 are of the same order of magnitude, B_1^2 is determined by the power of the rf source or by the maximum rf power tolerable without heating the sample. Thus, the method is limited to certain samples with long enough T_1, T_2, and τ (typically longer than nanoseconds). It requires also $|g^*|$ to be reasonably large, otherwise the conduction electrons will not couple to the rf field. This proves to be a limitation for $\mathrm{Ga}_{1-x}\mathrm{Al}_x\mathrm{As}$ alloys where g^* goes through zero when varying alloy composition.

Up to now, we described a situation where we directly observe the recombination of the conduction electrons, that is when the recombination line is due to band-to-band or band-to-acceptor transitions. Very often, in pure samples and at low temperature, the recombination light is due to excitonic transitions. Nevertheless, in our experiments, the optically detected resonance is that of the conduction electrons: this is evidenced in GaAs where the resonance line occurs at the same field, independently of the luminescence line under observation. This is because the saturation factor is significant only for the longest step of the process leading to recombination, that is the step during which the electrons are free before getting bound into excitons.[25]

C. Nuclear effects

It is well known that, in conditions of optical pumping at low temperature, it is possible to dynamically polarize the nuclei of the lattice.[17] When the relaxation of the nuclei is only due to the hyperfine interaction with the electrons, the mean nuclear spin $\langle I_{z\alpha}\rangle$ of species α is given by[26]

$$\frac{\langle I_{z\alpha}\rangle - I_{0\alpha}}{I_\alpha(I_\alpha+1)} = \frac{\langle S_z\rangle - S_0}{S(S+1)}\,, \tag{5}$$

where $I_{0\alpha}$ and S_0 are the thermal equilibrium values of the nuclear and electronic spins I_α and S; $\langle S_z\rangle$ is the mean electronic spin. This equation is valid for small polarizations, which is the case in all our experiments.

Under optical pumping conditions, in a longitudinal magnetic field, $\langle S_z\rangle$ is given by[27]

$$\langle S_z\rangle = \tfrac{1}{2}P = \frac{1}{2}\left(-0.5\frac{T_1(B_z)}{T_1(B_z)+\tau} + \frac{\tau}{T_1(B_z)+\tau}P_0\right), \tag{6}$$

where P_0 is the thermal equilibrium polarization of electrons in the magnetic field B_z:

$$P_0 = th\, g^*\mu_B B_z/2kT \approx g^*\mu_B B_z/2kT\,.$$

The nuclear polarization calculated from (5) and (6) is very large compared to usual thermal nuclear polarizations and it can have sizeable effects on the electrons. In particular, due to the hyperfine contact interaction, a nucleus α exerts on an electron a nuclear field $B_{n\alpha}$ given by[28]

$$\vec{B}_{n\alpha} = \tfrac{8}{3}\pi(\mu_0/4\pi)(g_0/g^*)\gamma_{n\alpha}\hbar\,|\psi(0)|^2\langle\vec{I}_\alpha\rangle\,, \tag{7}$$

where $\gamma_{n\alpha}$ is the gyromagnetic factor of the nucleus and $|\psi(0)|^2$ the modulus of the electronic wave function at the nucleus site. The total nuclear field B_N due to all the polarized nuclei is calculated[29] to be of the order of several teslas in GaAs for a full electronic polarization. In our experiments, although the electronic polarization is only about one percent, this nuclear field still has a drastic effect on the CESR. Instead of occuring in a well defined magnetic field $B_z = B_0$, the resonance takes place in the field $B_0 = |\vec{B}_z + \vec{B}_N|$, in which B_N is not precisely known.

It is then mandatory to suppress such nuclear fields to perform a meaningful measurement of B_0. From Eqs. (5) and (6), one sees that the nuclear and electronic polarizations originate from both optical and thermal orientations: the optical pumping contribution to B_N can be cancelled by modulating the circular exciting beam faster than the nuclear-spin-relaxation time T_{1N}: the optically created polarization averages to zero, as the nuclei are submitted to an average zero optical electronic polarization. This is evidenced in Fig. 1, which shows the giant shift of the CESR line due to the polarized nuclei: the nuclei have been left to attain their maximum polarization with a fixed exciting circular polarization. At time t_0, this fixed circular polarization is alternately modulated with either sign at a frequency of 50 kHz. The magnetic field is then swept through resonance at different time intervals. As the optical pumping component of the nuclear field B_N relaxes towards zero in the time T_{1N}, the resonance line shifts towards smaller values of $\vec{B}_N$. At very long times, it appears that in such conditions there still exists a small nuclear field, probably due to the thermal contribution in Eq. (6). To suppress this residual nuclear field, we perform a simultaneous saturation of the nuclear resonances to destroy the nuclear polar-

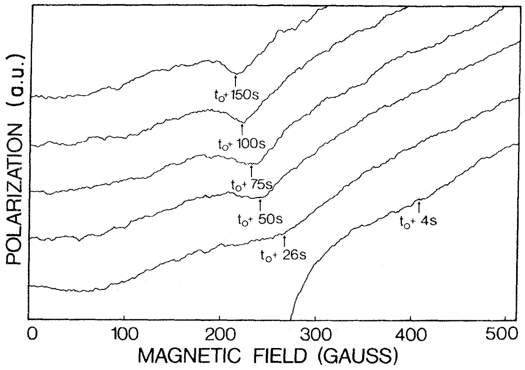

FIG. 1. Nuclear shift of the optically detected CESR line in GaAs. The nuclei were left to attain their equilibrium value under fixed circularly polarized excitation. At time t_0, this excitation polarization is modulated and the optically created nuclear polarization relaxes towards zero. The magnetic field B_z is scanned at the different times indicated. The successive curves were shifted upwards for clarity.

ization. In such conditions no nuclear field does exist in the sample and the electronic resonance occurs in the externally applied magnetic field $\vec{B}_0$. In the conditions of Fig. 1, the nuclear saturation was not performed and the asymptotic value of the resonance field is ≈220 G. This is different from the curve given in Fig. 3 where the nuclear saturation is performed and the resonance occurs at $B_0 = 250 \pm 10$ G. This difference of ≈30 G is the cause of the discrepancy between the g^* value reported here, $g^* = -0.44 \pm 0.02$ and the former value $|g^*| = 0.51$ given in a preliminary report.[30] Let us add that one should be very cautious when interpreting data in experiments involving light and magnetic fields since such internal nuclear fields should be present in many experimental situations of magneto-optics.

III. EXPERIMENTAL

The setup consists of a standard photoluminescence experiment at helium temperature adapted to measure circular polarizations and provide rf irradiation of the sample in a longitudinal magnetic field as depicted in Fig. 2. The exciting light source is that of a krypton laser tuned on the line which has its energy nearest to the band-gap energy. The longitudinal field is swept and the rf frequency is taken for conveniency at 151 MHz, as the rf field is simply produced by a loop placed around the Dewar tail. This setup delivers about $(1\text{G})^2$ of rotating field per watt of rf power. The limitation to the detectable $\Delta P/P$ being set by the signal-to-noise ratio attainable on the polarization, we were able to observe the resonance in samples of linewidths up to 30 G for our maximum rf power of 50 W. The resonance magnetic field B_0 is calibrated by the resonance of a DPPH (α, α'-diphenyl-β-picryl-hydrazyl) sample at 411 MHz.

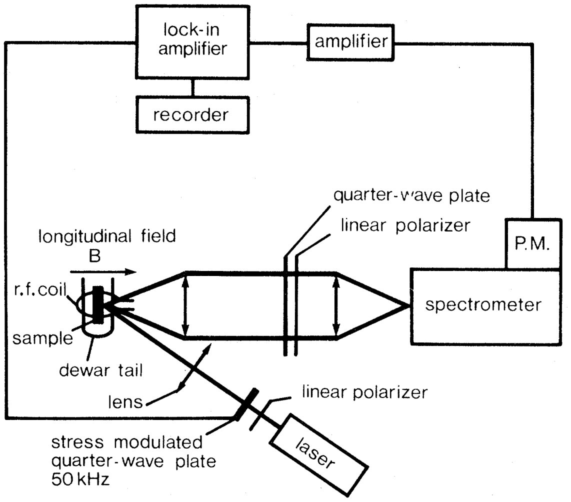

FIG. 2. Experimental setup for optical detection of CESR. The rf field at 151 MHz is produced by a single loop placed round the glass Dewar tail.

The absolute value of the polarization is measured in the following way: the polarization signal $L_+ - L_-$ is obtained by modulating the exciting light polarization and detecting one circular component of the luminescence light with a lock-in amplifier. The ratio of this signal with the total luminescence signal $L_+ + L_-$ gives $\mathcal{P}$. Whenever a definite value of $\mathcal{P}$ is not needed, as in detecting the CESR, we just record the signal $L_+ - L_-$. As mentioned above, we simultaneously perform the NMR of the lattice nuclei, ^{69}Ga, ^{71}Ga, ^{75}As and ^{113}In, ^{115}In, or ^{27}Al nuclei of alloys. This is done by submitting the sample to a resonant audiofrequency field sufficient to saturate the nuclear polarization: a coil (not represented in Fig. 2 for the sake of simplicity) is sequentially supplied with the resonance frequencies corresponding to the various nuclei of the lattice. These frequencies are swept along with B_z, so that the nuclei are always at resonance and saturated.

For CESR experiments, a careful selection of samples is necessary because of the need of sufficiently long T_1's, T_2's, and τ's. Thus the resonance of bulk doped p-type GaAs samples was not measured, as in such samples T_{2*} is of the order of[21] 10^{-11} sec, as compared to $T_{2*} \approx 10^{-8} - 10^{-9}$ sec in pure epitaxial samples.[25]

The GaAs sample used here is a vapor-phase epitaxy layer with an uncontrolled impurity concentration $|N_A - N_D| = 6 \times 10^{11}$ cm^{-3}. Its photoluminescence has been studied in great detail and is characteristic of very pure crystals.[25]

Ga$_{1-x}$Al$_x$As samples are liquid-phase epitaxy layers with a doping level of germanium acceptors of $\approx 10^{16} - 10^{17}$ cm^{-3}. Their energy gaps and compositions were determined from the position of the main luminescence peak, using the data of Shah *et al.*[31] The measured samples had $x = 0.12$; 0.26; 0.31; 0.36. We could not detect the CESR of samples with x between 0 and 0.26 because the few samples that we investigated had too small absolute values of g^*. It was however possible to estimate the g factor of Ga$_{0.88}$Al$_{0.12}$As by using a nonresonant method involving the evaluation of the hyperfine nuclear field[32]: from Eq. (7), one sees that the nuclear field becomes extremely large for small values of g^*. This nuclear field can be estimated from the oblique Hanle effect,[33] yielding a g^* value smaller than 0.05 for this sample. Although in this case the relative uncertainty is very large, the precision on $g^* - 2$ is very good.

$Ga_{1-x}In_xAs$ samples are liquid phase epitaxial layers with an uncontrolled impurity level of $\approx 10^{15}-10^{16}$ cm^{-3}. The exciting light energy is 1.55 eV. As mentioned above, with increasing x, we excite electrons with a larger kinetic energy in the conduction band. This leads to smaller polarizations and sets the limit to the indium content of samples at ≈10%. The indium content was determined by the measurement of the lattice constant through x-ray diffraction. Our samples had $x = 0.01$; 0.063; 0.08; 0.094.

IV. RESULTS AND DISCUSSION

Figure 3 displays some typical resonance curves. The upwards baseline observed on some samples is due to the variation of $\mathcal{P}$ with B_z because of an increase of the spin-relaxation time $T_1(B_z)$. It was verified that the linewidth of the CESR line is approximately equal to the transverse depolarizing field ΔB as can be predicted from Eqs. (3) and (4) (in the weak saturation regime $s \ll 1$). The most remarkable experimental fact is that this is true even in alloys. It shows that alloy disorder does not induce significant line broadening, which might have come from a distribution of the g values.

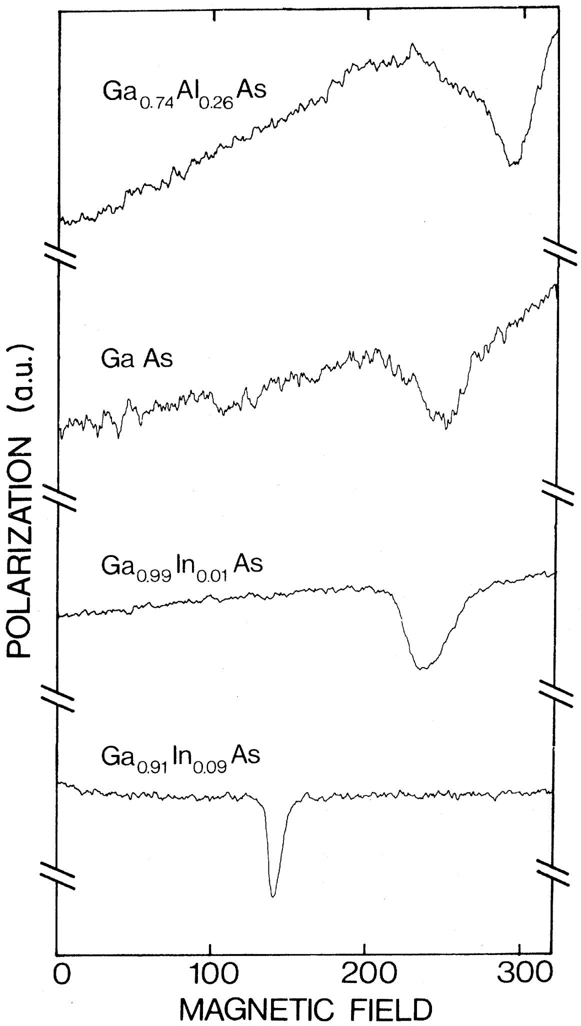

FIG. 3. Optically detected CESR lines for GaAs, $Ga_{1-x}In_xAs$, and $Ga_{1-x}Al_xAs$ at 1.7 K (rf frequency 151 MHz; field in gauss).

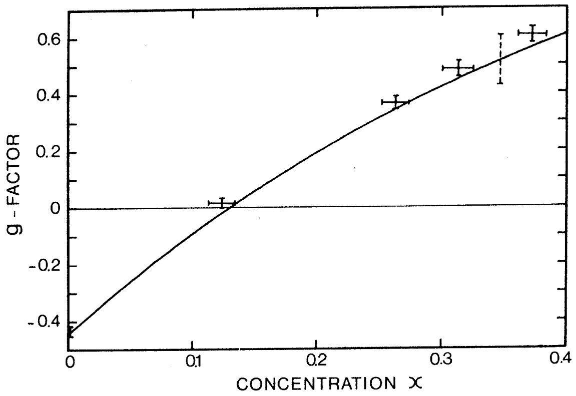

FIG. 4. g factor of $Ga_{1-x}Al_xAs$ alloys. The points indicated are experimental. The value for $x = 0.12$ is deduced by a nonresonant method (see Ref. 32). The curve is a theoretical calculation using multiband perturbation theory (see Ref. 2). The uncertainty bar of the curve comes from the uncertainty existing on the bowing parameter of the band-gap formula for the alloy (see Ref. 2).

Figure 4 and 5 show the results obtained for the alloys. The theoretical plot is the result of the multiband $\vec{k}\cdot\vec{p}$ perturbation calculation presented in Ref. 2.

Our results in alloys unambiguously give the sign of the g^* factors. Let us first recall that CESR with a conventional linearly polarized rf field does not permit to determine the sign of g factors, as

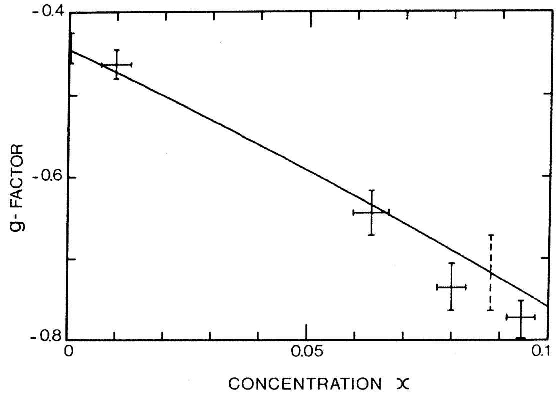

FIG. 5. g factor of $Ga_{1-x}In_xAs$ alloys.

it does not discriminate between the directions of precession of the spins. It only yields the absolute values of the g factors. But if we change the value of g^* in a known way, the change of the absolute value of g^* determines the sign of g^*. To predict the sense of variation of g^* with alloying, we can use at first order the three-band $\vec{k}\cdot\vec{p}$ perturbation theory[8]

$$g^*/g_0 = 1 - \tfrac{1}{3}P^2[1/E_0 - 1/(E_0 + \Delta_0)],$$

where P^2 is the interband momentum matrix element. The main effect of alloying is to change E_0. For instance, incorporating In in GaAs decreases the band gap, thus decreasing the algebraic value of g^*. As we measure by CESR an increase of the absolute value of g^*, it means that all g^*'s are negative. For $Ga_{1-x}Al_xAs$ alloys, the increase of the energy gap means an increase of g^* from its negative value in GaAs towards positive values. The increasing values of g^* are indeed observed by CESR for $x > 0.26$, but the decrease of $|g^*|$ at small x values could not be observed by CESR because of the smallness of g^*. All the sign determinations are independently confirmed by measuring the sign of $\vec{B}_N$ and using Eq. (7).[32]

Let us now compare our measurement of the g factor of GaAs : $g^* = -0.44 \pm 0.02$ with previous ones. The only previous resonant determination of g^* by standard EPR technique was performed by Duncan and Schneider.[7] They attributed a positive sign to their g factor $g^* = 0.5228$. The measurement was carried out in a quite highly doped sample $N_D \sim 5.10^{16}$ cm^{-3}. The usual nonresonant methods for determining g^*, i.e., high-field magneto-optical measurements on free carriers, could not for a long time yield a significant result for g^* because of its smallness, as was recognized by some authors.[34] It is the recent advent of high-purity samples with sharp reflectance or luminescence lines which permitted the measurement of g^* by analysis of the Zeeman splitting of various excitonic lines. White *et al.*[10] deduced the value $g^* = -0.43 \pm 0.05$ from the study of the Zeeman splitting of the exciton bound to the Sn acceptor. Nam *et al.*[35] measured $g^* = -0.50 \pm 0.05$ from the magneto-reflectance of the free exciton. Schairer *et al.*[36] give the value of -0.46 ± 0.02 in a more recent study of the Sn acceptor.

The only data significantly different from ours is that of Duncan and Schneider. The reason for this discrepancy is not understood at the moment. Let us point out, however, that their sample was quite heavily doped ($N_D \sim 5 \times 10^{16}$ cm^{-3}) as compared to ours.

V. CONCLUSION

The present determination of $g^* = -0.44 \pm 0.02$ in GaAs has the advantage over others of being resonant, thus precise, and unambiguous: it is the resonance of photocreated electrons in a high-purity material. No interpretation is required to link the measured data to g^*. The sign is also unambiguously measured. Let us stress the remarkable usefulness of the optical detection of CESR in such a case where the smallness of g^* makes precise determinations difficult. The measurement of the g^* factors in alloys has a great importance as it provides an important test of the validity of $\vec{k}\cdot\vec{p}$ theory, questioned up to now.[2] The CESR measurements confirm that spin properties are little or not affected by alloy disorder. This is to be compared with the same behavior of optical and transport properties.

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