

# Spin-resonance determination of the electron effective $g$ value of $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$

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The effective-spin splitting of the electrons at the bottom of the conduction band in bulk  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  was experimentally determined using two different spin-resonance techniques, optically detected spin resonance and electron paramagnetic resonance. The narrow linewidth, caused by motional narrowing, made a study of the Overhauser effect possible. The nuclear contributions from In, Ga, and As were identified by their relaxation times, and the experimental conditions necessary to avoid the build up of a nuclear magnetic field were established. The effective  $g$  value of  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  is  $g^* = -4.070 \pm 0.005$ . [S0163-1829(96)02336-3]

## I. INTRODUCTION

The effective spin-splitting factor  $g^*$  is a fundamental parameter of the electronic band structure in semiconductors, and can be used to test sensitively theoretical models of the band structure.<sup>1,2</sup> Such models for bulk semiconductors are essential for making correct predictions of optical and transport properties of low-dimensional structures. Even though the  $g$  values have been investigated in great detail for some binary semiconductors such as GaAs,<sup>3-5</sup> they are not known accurately for most ternary semiconductors such as the scientifically and technologically important material  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ .

Numerous attempts to determine experimentally the effective spin splitting factor  $g^*$  in  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  have been made, but the values scatter considerably.<sup>6-11</sup> Indirect measurements, where  $g^*$  is one of several fitting parameters, suggest  $g^* = -3.38$  (Ref. 6) or  $g^* = -4.5$ .<sup>7</sup> Magnetotransport measurements of two-dimensional electron gases (2DEG's) give spin-splitting values in the range 4.5–9.<sup>8</sup> Such measurements are, however, influenced by many-body effects such as spin exchange, and an “enhanced”  $g$  value is observed. These methods can therefore not be used to obtain reliable data for the “single-particle”  $g^*$  at the conduction-band edge.

The most direct way to measure  $g^*$  is by spin-resonance (SR) methods. Measurements reported so far for  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  are electron spin resonance detected in the magnetoresistivity in a 2DEG,<sup>9</sup> and electric dipole-induced SR observed in cyclotron-resonance experiments.<sup>10</sup> However, in both cases the spin splittings were magnetic field dependent, and did not give the effective  $g$  value of the conduction-band edge at low magnetic fields directly. In Ref. 9 the states involved in the high magnetic-field measurements are those at the Fermi energy of the 2DEG. Therefore, the measured spin splitting is for states high up in the conduction band. In Ref. 10 the  $g$  values (between  $-3.6$  and  $-3.8$ ) determined at high magnetic fields (between 10 and 16 T) were fitted by a  $\mathbf{k}\cdot\mathbf{p}$  calculation with several adjustable parameters to give the low-field value of  $g^*$ . The authors of Refs. 9 and 10 suggest values in the range  $-4.1 < g^* < -4.0$ . With the exception of one paper<sup>11</sup> that will be commented on below, no direct determination of  $g^*$  in bulk  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  has been reported to date.

In the present paper we report measurements of  $g^*$  on four  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  samples by two different spin-resonance techniques, optically detected spin resonance, and electron paramagnetic resonance (EPR). In these measurements,  $g^*$  is obtained directly from experiment using

$$E_{\mu w} = g^* \mu_B B_{\text{res}}, \quad (1)$$

where  $E_{\mu w}$  is the microwave energy, and  $\mu_B$  is the Bohr magneton.  $B_{\text{res}}$ , the magnetic field for resonant spin transitions, is smaller than 0.5 T. This is well below the high magnetic-field regime discussed above. Thus the value obtained from our measurements directly gives the *real* band-structure parameter  $g^*$ .

A possible systematic uncertainty in SR measurements is the displacement of the magnetic resonance due to magnetic fields other than the field  $B_0$  applied externally.<sup>3</sup> Such a magnetic field is the nuclear magnetic field  $B_N$  arising from a nonvanishing nuclear-spin polarization  $\langle I_z \rangle$  (Overhauser effect).<sup>12</sup> The field causes the resonance in the electron-spin system to occur at a field  $B_{\text{res}} = B_0 + B_N$ . If  $B_N$  is unknown, the value obtained for  $g^*$  might be erroneous. In the present work the Overhauser shift is studied systematically by high-precision EPR experiments. Conditions under which the nuclear magnetic field is absent were found, thus making an accurate determination of  $g^*$  possible. The result of our investigation is a precise effective  $g$  value for  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ :  $g^* = -4.070 \pm 0.005$ .

The paper is organized as follows. In Sec. II we will give a detailed description of the samples and the spin-resonance experiments. The spin-resonance results are presented and discussed in Sec. III A, the Overhauser shift measurements in Sec. III B, and a discussion of the implication of the result for the  $\mathbf{k}\cdot\mathbf{p}$  theory is given in Sec. III C. The conclusions follow in Sec. IV.

## II. EXPERIMENT

The samples were nominally undoped,  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  epilayers grown lattice matched by liquid-phase epitaxy (LPE) on a semi-insulating InP:Fe substrate. Details of the growth procedure can be found elsewhere.<sup>13</sup> The layer thickness was between 2 and 3  $\mu\text{m}$ . Hall measurements at room temperature showed free-electron concentrations from  $5.0 \times 10^{14}$  to

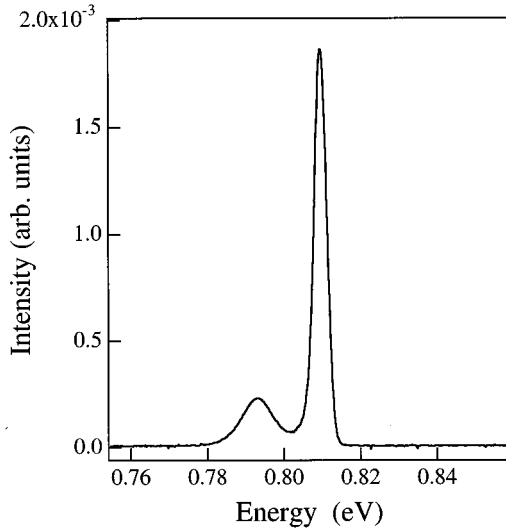


FIG. 1. Photoluminescence of LPE-grown  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  (sample TO27) at  $T=1.6$  K and an excitation power of  $P_{\text{laser}} \approx 100$  mW.

$2.0 \times 10^{15} \text{ cm}^{-3}$  and 77-K mobilities of 37 000–44 000  $\text{cm}^2/\text{V s}$ .

For the optically detected spin-resonance measurements, the photoluminescence (PL) was excited by 514-nm laser light with a power density of about  $100 \text{ mW}/\text{cm}^2$ . The PL was dispersed with an  $f/0.22$ -m double-grating monochromator, and detected with a Ge photoiode cooled to 77 K. The difference in intensity between the left ( $\sigma^-$ ) and right ( $\sigma^+$ ) circular-polarized components of the PL ( $I_{\sigma^-} - I_{\sigma^+}$ ) was analyzed by a photoelastic modulator operating at 42 kHz, and a linear polarizer placed in front of the monochromator. The samples, immersed in pumped liquid helium at  $\sim 1.6$  K, were located in an open 24-GHz  $\text{TE}_{011}$  cylindrical microwave cavity, itself placed at the center of a 4-T split coil superconducting magnet. About 100 mW of microwave power was applied. ( $I_{\sigma^-} - I_{\sigma^+}$ ) of the PL peaks was recorded in a Faraday configuration while scanning the magnetic field. Here the monochromator was used as a bandpass filter with a bandwidth of 2–5 meV. Optically detected spin-resonance measurements were also attempted by monitoring the absolute PL intensity, and using a  $p$ - $i$ - $n$  diode for chopping the microwaves at different frequencies. Standard lock-in techniques were used to record the various types of modulated signals.

The EPR experiments were performed in a Bruker ESP 300 spectrometer using a microwave frequency of 9.5 GHz. A proton resonance magnetometer was used to determine the magnetic field exactly. The samples were cooled to  $\sim 4.5$  K using a He gas flow cryostat.

### III. RESULTS AND DISCUSSION

#### A. Optically detected spin resonance and electron paramagnetic resonance

A typical PL spectrum from one of the lattice-matched  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  samples (TO27) is shown in Fig. 1. The main peak at an energy of 0.810 eV and with a full width at half maximum of 4 meV originates from unresolved free and

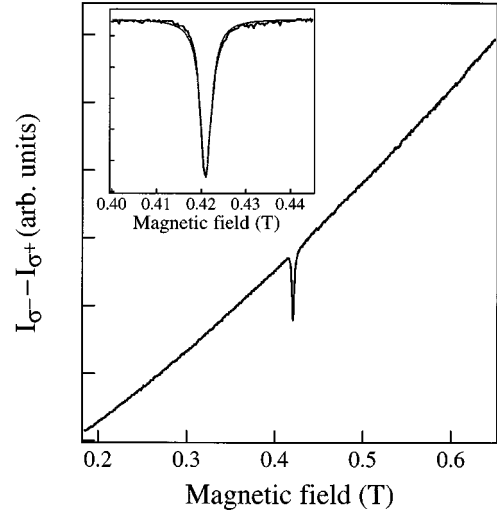


FIG. 2. Optically detected spin resonance ( $f_{\mu w} = 24$  GHz,  $T = 1.6$  K) detected on the magnetic circularly polarized excitonic PL of bulk  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  (sample TO27). The direction of the magnetic field is parallel to the growth axis. The inset shows the resonance after subtraction of a linear background. The shaded line represents a Lorentzian fit.

bound excitonic recombinations. Taking the excitonic binding energy into account, the peak energy corresponds to the band gap of lattice-matched  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ .<sup>14</sup> The broad PL peak at 0.79 eV is attributed to donor-acceptor pair recombination involving Zn as the acceptor.<sup>13</sup>

The optically detected spin-resonance spectrum detected on the excitonic PL peak of sample TO27 is shown in Fig. 2. The sharp resonance at  $B = 0.421$  T has a linewidth of 3.5 mT (see the inset in Fig. 2) and corresponds to a  $g$  value  $|g^*| = 4.08$  in the spin  $S = \frac{1}{2}$  formalism [Eq. (1)]. The  $g$  value is found to be negative and isotropic. The experimental error in the  $g$  value is  $\pm 0.02$ . Table I contains the values of  $g^*$  for the samples investigated.

The observation of SR by monitoring the circular polarization of the exciton PL is explained by the properties of the conduction- and valence-band states. In  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  the conduction band has an  $s$ -like character represented by  $|S, M_S\rangle = |\frac{1}{2}, \pm \frac{1}{2}\rangle$ , and the valence-band maximum is fourfold degenerate with heavy-hole states  $|J, M_J\rangle = |\frac{3}{2}, \pm \frac{3}{2}\rangle$  and light-hole states  $|J, M_J\rangle = |\frac{3}{2}, \pm \frac{1}{2}\rangle$ . Optical transitions with circular polarization  $\sigma^+$  ( $\sigma^-$ ) are for heavy-hole states allowed between  $M_S = -\frac{1}{2}$  and  $M_J = -\frac{3}{2}$  ( $M_S = +\frac{1}{2}$  and  $M_J = +\frac{3}{2}$ ), and for light-hole states between  $M_S = +\frac{1}{2}$  and  $M_J = -\frac{1}{2}$  ( $M_S = -\frac{1}{2}$  and  $M_J = +\frac{1}{2}$ ). The probability for light-hole tran-

TABLE I. The experimental values of  $g^*$  for the different samples.

Sample	$g^*$ from optically detected SR	$g^*$ from EPR	$n$ at 300 K ( $\text{cm}^{-3}$ )
TO23	$4.07 \pm 0.02$	$4.074 \pm 0.005$	$2.0 \times 10^{15}$
TO27	$4.08 \pm 0.02$	$4.065 \pm 0.005$	$1.8 \times 10^{15}$
TO28	$4.07 \pm 0.02$	$4.070 \pm 0.005$	$5.0 \times 10^{14}$
TO29	$4.06 \pm 0.02$		$1.1 \times 10^{15}$

sitions is, however, three times smaller than for heavy-hole transitions.<sup>15</sup> Thus  $M_S = -\frac{1}{2}$  ( $+\frac{1}{2}$ ) conduction-band electrons preferentially recombine by  $\sigma^+$  ( $\sigma^-$ ) emission. This is also true for electrons and holes bound as excitons. A magnetic field will lift the spin degeneracy ( $M_S = \pm\frac{1}{2}$ ) of the conduction band, and, if the electrons can thermalize before optical recombination, they will preferentially populate the lowest Zeeman split states. Therefore, microwave quanta are absorbed at the resonant magnetic field [Eq. (1)], thus equalizing the population of the spin-up and spin-down states. Since the two spin states recombine with opposite circular polarization it is possible to observe SR of the conduction-band electrons as a resonant decrease in the  $(I_{\sigma^-} - I_{\sigma^+})$  signal. Because of the spin conservation during the formation of excitons, this description also holds for exciton recombination.<sup>16</sup> In the present case, the assignment of the SR to free electrons is based on the fact that the presence of free electrons is the step with the longest time constant in the process: excitation—thermalization of electrons and holes—formation of excitons—their recombination.<sup>16</sup> A spin flip of electrons is simply more probable than a spin flip of excitons.

A clear experimental verification of the thermalization is the observation that  $(I_{\sigma^-} - I_{\sigma^+})$  increases almost linearly with magnetic field (Fig. 2).<sup>17</sup> Furthermore, the absence of a SR signal when detecting the total PL is consistent with the fact that SR alters the distribution of the carriers in the different spin states, but not the total number of electrons involved in the radiative recombination. The influence of hole spins on the polarization is disregarded because of the very short hole spin relaxation times. In bulk GaAs the hole spin-relaxation time is in the picosecond range.<sup>17</sup> A similar short hole spin-relaxation time is assumed here for  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ . This should be compared to the radiative lifetimes and electron spin-relaxation times which are more than one order of magnitude larger.

An EPR spectrum of sample TO23 with the magnetic field directed along the sample growth direction is shown in Fig. 3. The sharp resonance line allows an accurate determination of  $g^*$  of the conduction-band electrons in  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ . The results for our samples are collected in Table I. The error in the determination of  $g^*$  is  $\pm 0.005$ .

For sample TO23 the resonance line has a peak-to-peak width of 0.33 mT. The width increases with decreasing carrier concentration to 1 mT for the sample with the lowest carrier concentration. Such a dependence of the linewidth on carrier concentration is characteristic of motional narrowing of the resonance line, as described by Clerjoud *et al.*<sup>18</sup> for conduction-electron spin resonance in bulk InP. For the samples that had a carrier concentration as low as our samples, they found similar EPR linewidths, and argued that the motional narrowing is due to thermally activated hopping of donor electrons between different donor sites. In the present study the optically detected SR results give evidence that free electrons are responsible for the observed signals. However, in EPR experiments performed with illumination of the sample, no clear change of the signal intensity could be observed. This can be explained by the spin resonance of electrons that are bound to shallow donors. The spin splitting obtained in this way is, however, virtually identical to that of free conduction-band electrons, since the shallow donor

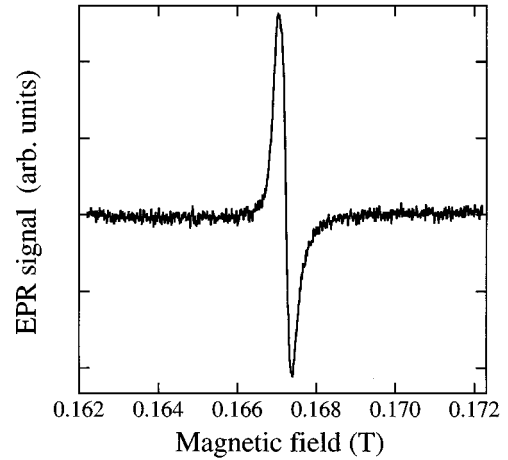


FIG. 3. An EPR spectrum ( $f_{\mu w} = 9.5$  GHz,  $T = 4.5$  K) of bulk  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  (sample TO23).

states are derived from states close to the conduction-band minimum. Regardless of the origin of the spin resonance, free or donor-bound electrons, the sharp resonance lines observed in the present EPR experiments can be explained by motional narrowing.

For some samples the resonance position was found to shift slightly with varying direction of the magnetic field. This shift was always smaller than half a percent, and showed an axial symmetry corresponding to a  $g$  tensor with its main axes parallel and perpendicular to the sample growth direction. We attribute this tiny anisotropy to a small lattice mismatch between the LEP grown ternary layer and the InP substrate.<sup>2</sup> This assumption is supported by PL results that showed small variations in the near-band-gap luminescence for those samples.

The value  $g^* = -4.070 \pm 0.005$  obtained from the EPR measurements is in excellent agreement with  $g^* = -4.07 \pm 0.02$  determined by the optically detected spin-resonance measurements (see Table I).  $g^* = -4.070 \pm 0.005$  is the most exact effective  $g$  value of the conduction-band electrons in  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  that has been published to date. It is in agreement with  $|g^*| \approx 4.1$  as obtained from an extrapolation of high magnetic-field spin-resonance data.<sup>9</sup> It is also in reasonable agreement with the extrapolated values of  $|g^*| = 4.04$  or  $|g^*| = 4.08$  from Ref. 10, and in good agreement with our prediction from an earlier study.<sup>19</sup> It is, however, very different from  $|g^*| = 5.2$ , as was obtained in the first spin-resonance experiments by Kana-ah *et al.*<sup>11</sup> The mechanisms leading to the optically detected magnetic-resonance results observed for a single sample in Ref. 11 were not discussed, making the assignment to the conduction electron  $g$  value questionable.

## B. Overhauser shift

A possible source of error in our  $g^*$  measurement is the Overhauser shift.<sup>12</sup> This effect causes the resonance in the electron-spin system to occur at a field  $B_{\text{res}} = B_0 + B_N$ . We have investigated this effect in order to establish the experimental conditions for the correct determination of  $g^*$ .

The nuclear magnetic field has its origin in a nonvanishing nuclear-spin polarization  $\langle I_z \rangle$ , and is given by<sup>12</sup>

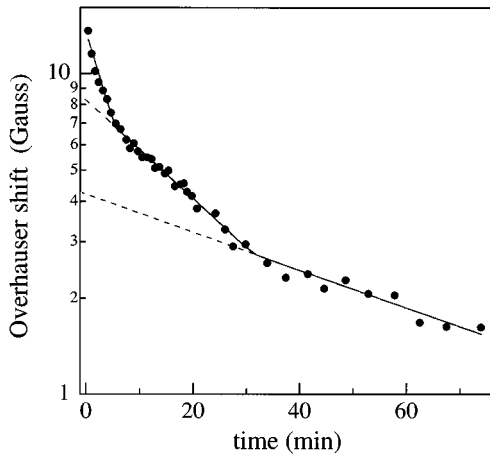


FIG. 4. The Overhauser shift of the EPR line obtained for  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  (sample TO23) after dynamic nuclear polarization. The solid line is the result of a triple-exponential fit.

$$B_N = \frac{A}{g^* \mu_B} \langle I_z \rangle, \quad (2)$$

where  $g^*$  is the effective electron  $g$  value, and  $A$  is the coupling constant of the Fermi contact interaction, or hyperfine interaction,  $\text{AIS}$  of the nuclear and electron spins. A preferential nuclear polarization can be introduced by dynamic nuclear polarization (DNP), i.e., electron-spin relaxation flipping the nuclear spins by flip-flop processes induced by the interaction  $\text{AIS}$ . Such spin relaxation is substantial, when the electron-spin distribution returns to thermal equilibrium after resonant absorption of microwave quanta with saturating power densities.

In the present EPR experiments on bulk  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ , the Overhauser shift could deliberately be introduced by DNP in a slow-passage magnetic-resonance experiment following the scheme employed in Ref. 18. A nuclear polarization was built up by fulfilling the magnetic-resonance condition using a high microwave power ( $P_{\mu w} \approx 100$  mW) for about 25 min. A 1.5-mT shift of the magnetic resonance away from the initial resonance position was observed. This shift is small compared to a similar DNP experiment on InP, which can be explained by the difference in electron concentration.<sup>18</sup> For the subsequent rapid scan measurements the microwave power was reduced to  $P_{\mu w} \approx 200$   $\mu\text{W}$  in order not to alter the nuclear polarization. Because of the unusually narrow EPR linewidth, it was possible to measure the decay of the nuclear polarization by monitoring the time evolution of the resonant magnetic field. According to Eq. (2),  $B_N$  decreases with decaying  $\langle I_z \rangle$ . A plot of the Overhauser shift as a function of time shows an exponential decay with three time constants (Fig. 4). The time constants are 235 s, 14 min, and 68 min. The different time constants show that several spin subsystems originating in the different nuclear species in  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  are involved. It is illuminating to compare these time constants with nuclear-spin-relaxation times reported in the literature. For In in InP, with the isotopes,  $^{115}\text{In}$  and  $^{113}\text{In}$  in the abundance ratio 95.7:4.3, values for the spin-relaxation time  $T_1^{(N)} = 450$  and 209 s are reported.<sup>18,20</sup> This agrees with the fastest decay time found in our experiment. For Ga in GaAs, with  $^{69}\text{Ga}$  and  $^{71}\text{Ga}$  in the

abundance ratio 60.1:39.9, the average nuclear-spin-relaxation time is  $T_1^{(N)} = 67$  min,<sup>4</sup> in agreement with the longest time constant in our experiment. In the same work the  $^{75}\text{As}$  nuclei (100% abundance) show  $T_1^{(N)} = 16$  min, which is close to our medium fast decay time.<sup>4</sup> In our quantitative analysis the different hyperfine coupling constants of the three nuclear species have not been considered.<sup>4</sup> However, the triple-exponential decay and the time constants found here justify an assignment of the EPR shift to the Overhauser effect originating in the different nuclear species in  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ .

The important consequence of this work is that the Overhauser effect can be controlled. In particular, in our EPR experiments the Overhauser effect could be avoided if the microwave power was small compared to  $P_{\mu w} \approx 100$  mW, and the external magnetic field was swept rapidly from zero to the resonance position. Under these experimental conditions the value of  $g^*$  is determined only by the external magnetic field.

### C. Calculation of $P^2$ and $P'^2$

In III-V semiconductors,  $\mathbf{k} \cdot \mathbf{p}$  theory is found to model the band structure at the bottom of the conduction band (the  $\Gamma$  point) with high accuracy.<sup>1</sup> In the five-band approximation, the interband coupling is parametrized by the matrix elements  $P$  and  $P'$  and the constants  $C$  and  $C'$ . Here  $P^2$  and  $P'^2$  can be accurately determined, provided that  $g^*$  and  $m^*$  (the electron effective mass at the bottom of the conduction band) and the energies of the band extrema are known from experiments.  $P^2$  and  $P'^2$  are given by the well-known expressions derived in Ref. 1:

$$P^2 = A \left[ \left( 1 + C' - \frac{g^*}{g_0} \right) \left( \frac{2}{E_{\Gamma}^{8c} - E_0} + \frac{1}{E_{\Gamma}^{7c} - E_0} \right) - \left( 1 + C - \frac{m_0}{m^*} \right) \left( \frac{1}{E_{\Gamma}^{7c} - E_0} - \frac{1}{E_{\Gamma}^{8c} - E_0} \right) \right],$$

$$P'^2 = A \left[ \left( 1 + C' - \frac{g^*}{g_0} \right) \left( \frac{2}{E_0} + \frac{1}{E_0 + \Delta_0} \right) + \left( 1 + C - \frac{m_0}{m^*} \right) \left( \frac{1}{E_0} - \frac{1}{E_0 + \Delta_0} \right) \right],$$

$$A = \left( \frac{1}{E_0(E_{\Gamma}^{7c} - E_0)} - \frac{1}{(E_0 + \Delta_0)(E_{\Gamma}^{8c} - E_0)} \right)^{-1}. \quad (3)$$

For  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ , the energy differences are  $E_0 = E_{\Gamma}^{6c} - E_{\Gamma}^{8v} = 0.813$  eV,  $\Delta_0 = 0.36$  eV,<sup>21</sup>  $E_{\Gamma}^{7c} - E_{\Gamma}^{8v} = 4.44$  eV, and  $E_{\Gamma}^{8c} - E_{\Gamma}^{8v} = 4.62$  eV.<sup>22</sup> Using our value of  $g^*$ , an effective mass  $m^* = 0.041m_0$ ,<sup>10,21</sup> and  $C = 2$  and  $C' = 0.02$ ,<sup>1</sup> we obtain  $P^2 = 23.67$  eV and  $P'^2 = 17.75$  eV. It should be noted that already the three-band approximation in  $\mathbf{k} \cdot \mathbf{p}$  theory<sup>3</sup> gives an accurate value for  $P^2$ , i.e.,  $P^2 = 24.12$  eV.

#### IV. CONCLUSIONS

The conduction-band spin splitting in bulk  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  was experimentally determined using two different spin-resonance techniques, optically detected spin resonance and EPR. The narrow linewidth, caused by motional narrowing, made a study of the Overhauser effect possible. The nuclear contributions from In, Ga, and As were identified, and the experimental conditions to avoid the buildup of a nuclear magnetic field were established. The effective  $g$  value of  $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$  determined is  $-4.070 \pm 0.005$ . This results in

modified matrix elements in the five-band  $\mathbf{k} \cdot \mathbf{p}$  theory:  $P^2 = 23.67$  eV and  $P'^2 = 17.75$  eV.

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