High-field spin resonance of weakly bound electrons in GaAs

M. Seck, M. Potemski, and P. Wyder

High Magnetic Field Laboratory, Max-Planck-Institut für Festkörperforschung/Centre National de la Recherche Scientifique,

Boîte Postale 166, F-38042 Grenoble Cedex 9, France

(Received 28 April 1997)

Electron spin resonance (ESR) of shallow donor electrons in *n*-type GaAs has been observed by means of direct detection of microwave absorption at magnetic fields of 6–11 T. The ESR structure is smeared out over a magnetic field range of up to 1 T. The line shape is strongly asymmetric and depends on the magnetic-field sweep direction. These unusual features are assigned to microwave-induced nuclear polarization under ESR conditions, leading to strong effective nuclear fields (Overhauser shift). The ESR curves show a signature of nuclear magnetic resonance if an additional radio-frequency field is applied. The observed ESR line shape is well reproduced by numerical simulation. Furthermore, the Landé g factor of weakly localized electrons in GaAs has been accurately determined ($g = -0.464 \pm 0.002$ at B = 0). [S0163-1829(97)00636-X]

I. INTRODUCTION

Current knowledge of spin-dependent properties of conduction-band states in GaAs-based materials has been established essentially on the basis of optical pumping experiments,¹⁻⁴ the observation of cyclotron resonance splitting at high magnetic fields,⁵ and measurements of the dc conductivity response to microwave excitation of a twodimensional electron gas (2DEG).⁶⁻⁸ When combined with theoretical models, these methods permit a rough estimation of the corresponding g factor (-0.44 ± 0.02 in the bulk) and they have underlined the importance of the coupling of the electron spins to the nuclear spins (an effective hyperfine field of up to 5.3 T can be expected⁹). These conclusions are now generally accepted, although they remain in contradiction with early experiments reporting conventional detection (by microwave absorption) of electron spin resonance (ESR) in *n*-type GaAs.¹⁰ Although pure ESR methods usually give access to direct and precise information about spin properties of the investigated system, conventional ESR spectroscopy seems to be hardly applicable to GaAs since the expected resonance field falls out of the range of standard ESR spectrometers and their sensitivity may not be sufficient to detect the broad lines. GaAs is one of the most widely investigated and applied semiconductor compound, whereas the electronic g factor, an essential parameter to test the bandstructure model, remains experimentally rather poorly determined in this material. Physics of ESR in GaAs is expected to involve unique effects of dynamic nuclear spin polarization since the small value of the electronic g factor implies that the Zeeman energy is comparable with typical interaction energies between electronic and nuclear spins.

In this paper the direct observation of ESR in a series of n-type GaAs samples at high magnetic fields is reported. To our knowledge, this is the first ESR study of electrons bound to shallow donor sites in GaAs. A very unusual line shape is observed. This is assigned to the effect of dynamic nuclear polarization (DNP) as confirmed in ESR experiments under additional radio-frequency irradiation corresponding to conditions of nuclear magnetic resonance (NMR). The main features of the observed ESR response are understood qualita-

tively in terms of a model simulation, implying a typical longitudinal nuclear spin relaxation time in the second range. Moreover, the experiments allow us to determine precisely the Landé g factor of shallow donor electrons.

II. SAMPLES

A number of *n*-type GaAs samples, grown under different conditions and with different donor concentrations, have been examined. Clear and qualitatively the same ESR signals have been found at low temperatures (1.4-2.5 K) for several samples with relatively low concentration of neutral donors $(2.6 \times 10^{14} \text{ cm}^{-3} \leq N_D^0 \leq 2.7 \times 10^{15} \text{ cm}^{-3})$. These samples were either grown by liquid-phase epitaxy and unintentionally doped with residual sulfur donors or bulk ingot grown and intentionally doped with tellurium. Table I shows the parameters of the samples.

III. EXPERIMENTAL SETUP

The experiments have been performed using a sensitive broadband (40-60 GHz) ESR spectrometer combined with a superconducting magnet that supplies a maximum magnetic field of 16 T. The microwave sources are composed of a quartz-stabilized YIG (yttrium iron garnet) oscillator working at 10–15 GHz and an active frequency quadrupler with a solid-state amplifier. The microwaves are fed into a frequency tunable cylindrical cavity and the reflected power is measured by a phase-sensitive heterodyne detection system that allows for simultaneous detection of absorption and dispersion in the sample. We apply a magnetic-field modulation

TABLE I. List of the *n*-doped GaAs samples.

Sample	Growth technique	Carrier concentration $N_D - N_A \text{ (cm}^{-3}\text{)}$	n dopant
A	LPE	6.6×10^{14}	S (residual)
В	ingot	2.5×10^{15}	Te
С	ingot	6.5×10^{14}	Te
D	ingot	2.6×10^{14}	Te

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FIG. 1. Typical derivative ESR signal of shallow donors in GaAs obtained at T=1.4 K and 52.02 GHz microwave frequency from sample A. Data are shown at different microwave powers, where 0 dB corresponds to ≈ 1 mW at the cavity. χ'' and χ' are, respectively, the absorption and the dispersion part of the signal. The arrows indicate the sweep direction. Note that magnetic susceptibilities are presented (a linearly scaled ESR signal would show power-independent traces). The field modulation frequency and amplitude are 14 Hz and 6 mT peak to peak. The magnetic-field sweep rate is 0.3 T/min.

technique and measure derivative ESR lines. The microwave cavity is placed in a closed tube filled with helium exchange gas, with the ensemble installed in a helium bath cryostat, which in turn is situated in the room-temperature bore of the magnet. With this cryogenic setup, the temperature can be varied from 1.4 to 10 K with good stability. The sensitivity of the setup is between 10^8 and 10^9 spins per 0.1 mT of ESR linewidth.

IV. EXPERIMENTAL RESULTS

Figure 1 shows the ESR response of lightly doped n-type GaAs at different microwave powers. Surprisingly, the line shape is asymmetric and strongly depends on the magnetic-field sweep direction. However, it is reproduced exactly in subsequent field cycles. This behavior cannot be due to the well-known passage effects, as the time-reversal symmetry conditions for spin systems obeying the Bloch equations are not fulfilled.¹¹

In each sweep direction, the ESR response appears rather suddenly at a certain magnetic field (here 8.94 T) and afterward a decay of the signal is observed, which is broader at high microwave power. A remarkable feature is that the ESR susceptibility noticeably *decreases* at low microwave power,



FIG. 2. Dispersion signal from sample *A* in the presence of an additional radio-frequency field at three different frequencies. The vertical arrows indicate the NMR field of ⁷⁵As. The microwave frequency and power are 46.63 GHz and -12 dB. Temperature is 1.6 K. The NMR frequency/field ratio is 7.29 ± 0.03 MHz/T, in agreement with the literature value for ⁷⁵As (7.292 MHz/T).

in contrast to usual saturation effects. All the samples investigated show a very similar behavior.

It is important to note that in addition to the signal component in phase with the modulation field, there is also an out-of-phase component (not shown here) at a modulation frequency as low as 14 Hz. The out-of-phase signal becomes stronger if the modulation frequency is lowered. This indicates the presence of a slow process in the sample, with a characteristic time in the range of seconds.

One would expect that the signal shape should depend on the magnetic-field sweep rate. However, due to experimental restrictions (long-term stability of the setup and limited sweep rate of the superconducting magnet), we could vary the sweep rate only in the range between 0.05 and 0.5 T/min, where the signal shape remains almost unchanged.

The appearance of slow process suggests a contribution of nuclear spins to the observed ESR response. This is clearly evidenced in ESR measurements under additional radio-frequency irradiation corresponding to NMR conditions (see Fig. 2). At the NMR resonance field of ⁷⁵As, a jump or a dip in the ESR line is visible. Similar though weaker signatures of NMR have been observed when saturating the ⁶⁹Ga or ⁷¹Ga nuclei.

The dependence of the ESR signal on experimental parameters is very unusual. We have found that lowering the microwave power, increasing the temperature, and decreasing the microwave frequency and magnetic field have similar effects on the signal shape, which are to narrow the hysteresis structure and to decrease the intensity. Figure 3 shows the temperature effect.

V. INTERPRETATION

A. Qualitative picture

Most of the striking features of the ESR signal can be qualitatively understood. The scalar hyperfine interaction



FIG. 3. Temperature dependence of the ESR signal in sample A. The microwave frequency and power are 52.02 GHz and -12 dB. Increasing temperature has a similar effect as decreasing microwave power. The signal intensity decreases (more than expected from Curie law) and the width of the hysteresislike structure decreases.

 $AI \cdot S$ between nuclear spins I and electron spins S plays an essential role, but the hyperfine structure is not resolved, as the 1s wave function of a shallow donor electron in GaAs extends over $\sim 10^5$ nuclei (Bohr radius $a_B = 10$ nm, which shrinks¹³ to about 7 nm in a magnetic field of 10 T). However, when the nuclei are polarized, the hyperfine interaction shifts the ESR line by an effective nuclear field B_n (Overhauser shift). B_n reaches 5.3 T at full nuclear polarization.⁹

The essential point is that the nuclei become polarized by the microwaves, but only while the electron spins are in resonance, and the nuclear polarization in turn shifts the ESR resonance field (Overhauser shift). This interaction between line position and nuclear polarization leads to a situation where the ESR line moves synchronously with the external field over a certain range, once the initial resonance field has been crossed. The mechanisms of microwave-induced DNP are discussed later. So far, three main experimental features can be explained. (i) The asymmetric ESR signal shape is due to the microwave-induced Overhauser shift, which occurs only after crossing the initial resonance field (dashed vertical line in Fig. 1). (ii) High microwave power drives the nuclear polarization far out of thermal equilibrium, thus the ESR response is broader than at low power. (iii) If a nuclear species is saturated by NMR, the ESR signal tends to go to zero as the line snaps back towards its initial position. However, if the NMR occurs in the vicinity of the initial ESR line position (second curve in Fig. 2), the nuclear polarization recovers afterward and a dip is observed instead of a jump towards zero.

From these qualitative considerations, one can also understand why the ESR susceptibility decreases at low micro-



FIG. 4. Signal enhancement by DNP: The individual ESR lines (dotted) form an inhomogeneously broadened line (dashed), but by DNP and the Overhauser shift, the lines are accumulated at one position, corresponding approximately to the current field (solid line).

wave power: We believe that the width of the initial ESR line (before the field sweep) is at least several tens of mT due to inhomogeneous broadening caused by statistical orientation of nuclear spins as well as by fluctuations of the electron g factor.¹⁴ The homogenous linewidth of an individual electron is likely to be only a few mT. Medium to high microwave power accumulates the ESR lines of individual electrons, as all of them follow the external field. Thus DNP enhances the susceptibility and may even be necessary to make ESR observable. This effect is illustrated in Fig. 4.

This picture is confirmed regarding the behavior of the ESR signal when experimental parameters are varied: In Sec. IV we mentioned that lowering the microwave power, increasing the temperature, and decreasing the microwave frequency and magnetic field have similar effects on the signal, which are to narrow the hysteresis structure and to decrease the intensity. This indicates less effective DNP, which can indeed be expected. Higher temperature increases the nuclear and electronic spin relaxation rates and tends to destroy DNP. At lower magnetic fields, the increasing overlap of the donor wave function¹³ enables hopping of the electrons and the frequently changing nuclear surrounding may also oppose DNP. Efficient electron hopping is probably also the reason why no ESR is observed in samples with carrier concentrations above 10^{16} cm⁻³ and/or at temperatures above 3 K.

B. Electronic g factor

In spite of the large hysteresis structure, the electronic g factor can be determined with good precision when fixing the initial position of the ESR line in the center between the onset fields of the signal for the two sweep directions. Figure 5 shows the magnetic-field dependence of the g factor for different investigated samples. A common behavior is observed in spite of different sample parameters such as carrier concentration, growth process, and dopant nature. By extrapolation we obtain the zero field value of $g = -0.464 \pm 0.002$. It falls in the range of previously reported data for conduction electrons, 1-4 though the accuracy is remarkably increased. Our error specification is very conservative and corresponds to the difference between the two



FIG. 5. Field dependence of the electronic g factor of shallow donor electrons in GaAs. The negative sign has been taken over from literature (Ref. 12).

signal onset fields, thus including eventual systematic errors by choosing the center of this field range. An almost negligible correction due to thermal polarization of the nuclei also has been taken into account. The magnetic-field dependence of the *g* factor is found to be $0.0052\pm0.0005 \text{ T}^{-1}$, in agreement with recent measurements⁴ and with calculations for bulk GaAs⁵ and similar to values reported in a 2DEG for the lowest Landau level.⁷

C. Computer simulation

For a more quantitative description of the observed ESR line shape, we develop a simple model combining two wellknown mechanisms of DNP.^{15,16} (i) For the case of conduction electrons in metals or semiconductors, or in liquids, saturation of the allowed ESR transitions w_1 [see Fig. 6(a)] with $\Delta m_I = 0$ polarize the nuclei through flip-flop relaxation w_2 due to S_+I_- and S_-I_+ terms of the scalar hyperfine interaction $AI \cdot S$. (ii) For the case of fixed impurities, the anisotropic hyperfine interaction (here the dipole-dipole interaction) admixes the states $|m_S, m_I\rangle$ and $|m_S, m_I \pm 1\rangle$ by the amount α and thus enables forbidden ESR transitions w_2 and w_3 including a nuclear spin flip. Because of the additional nuclear Zeeman energy, the forbidden lines are satellites of the w_1 transition [Fig. 6(b)]. They are very effective in DNP, but too weak to be observed directly in the ESR response, as the transition probability is only $4\alpha^2$ compared to w_1 . We believe that the system of shallow donors in GaAs at low temperatures is an intermediate case of reduced electron mobility, allowing for both mechanisms of DNP.

In the numerical simulation we consider a system of 1000 electrons with individual ESR resonance fields B_0^i , distributed in a Gaussian shape with a full width at half maximum ΔB . Each electron *i* "sees" an individual effective nuclear field B_n^i that shifts the resonance to $B_0^i - B_n^i$. We assume that the ESR absorption line g(B) of a single electron is a Lorentzian:

$$g(B) = \frac{\gamma_e T_{2e}}{\pi \{ 1 + (T_{2e} \gamma_e B)^2 \}},$$
 (1)



FIG. 6. Model used for computer simulation of the line shape: (a) The allowed (w_1) and forbidden (w_2, w_3) ESR transitions between energy levels of an S=1/2 electron with negative g factor and a single I=1/2 nucleus (for simplicity) with a positive g factor. Further levels corresponding to different nuclei are indicated as "dashed levels." (b) ESR line positions at fixed frequency and the corresponding direction of the Overhauser shift induced by DNP.

where T_{2e} and γ_e are the transversal spin relaxation time and the gyromagnetic ratio of the electron, respectively. The observed ESR absorption signal is thus proportional to

$$\sum_{i} g(B - B_0^i + B_n^i).$$
 (2)

The problem is to determine the evolution of the B_n^i during a sweep of the external magnetic field *B*. In order to limit the number of unknown parameters, we describe the spatial distribution of the nuclear spin polarization $p(\vec{r})$ around an electron *i* centered at $\vec{r}=\vec{0}$ using a single variable p_i . The Overhauser shift is simply given by $B_n^i = -5.3 \text{ T} \times p_i$, if we choose p_i to be the weighted average of $p(\vec{r})$ over the volume of the electronic wave function $\psi(\vec{r})$:

$$p_i = \int |\psi(\vec{r})|^2 p(\vec{r}). \tag{3}$$

Now we assume for simplicity that the DNP of nuclear spins contributing to p_i can be described by an effective admixture coefficient α (dipole-dipole interaction) and by an effective nuclear spin relaxation rate $1/T_{1n}$ (via spin flip-flop processes including an electron spin flip). In the frame of this strongly simplified model, neglecting (i) the spatial dependence of the different DNP and spin relaxation mechanisms, (ii) different spin relaxation times of the nuclear species, (iii) nuclear spin diffusion, (iv) effects due to electron hopping between the donor sites, and (v) complications due to spin I=3/2 of the involved nuclei, we obtain the following rate equation for the p_i :



FIG. 7. Line shape obtained by numerical simulation based on Eq. (4), at a microwave power of -18 dB and -30 dB. Higher microwave power heats the sample and would require changes of the model parameters. Note the signal enhancement with respect to a simulation with DNP disabled (dotted).

$$\begin{aligned} \frac{dp_i}{dt} &= -\frac{1}{T_{1n}} \bigg\{ p - p_0 \bigg[1 - s \frac{\gamma_e}{\gamma_n} \bigg] \bigg\} \\ &+ \sum_q 2\pi \kappa_q \alpha^2 \gamma_e B_{1i}^2 \epsilon_T g \bigg(B - B_0^i \bigg[1 + \frac{\gamma_n^q}{\gamma_e} \bigg] + B_n^i \bigg) \\ &- \sum_q 2\pi \kappa_q \alpha^2 \gamma_e B_{i1}^2 \epsilon_T g \bigg(B - B_0^i \bigg[1 - \frac{\gamma_n^q}{\gamma_e} \bigg] + B_n^i \bigg). \end{aligned}$$

$$(4)$$

The subsequent terms on the right-hand side correspond to w_1 , w_2 , and w_3 transitions, respectively. Different nuclear species q with relative concentrations κ_q are distinguished, where q is ⁷⁵As, ⁶⁹Ga, or ⁷¹Ga and $\Sigma \kappa_q = 1$. p_0 is the nuclear polarization in thermal equilibrium and s is the ESR saturation parameter

$$s = \frac{\gamma_e^2 B_{1i}^2 T_{1e} T_{2e}}{1 + [T_{2e} \gamma_e (B - B_0^i + B_n^i)]^2 + \gamma_e^2 B_{1i}^2 T_{1e} T_{2e}}.$$
 (5)

The γ_n^q are gyromagnetic ratios of the nuclear species q, with the average value $\gamma_n = \sum_q \kappa_q \gamma_n^q$. T_{1e} and T_{2e} are the longitudinal and transversal electron spin relaxation times. B_{1i} is the microwave magnetic field in the cavity, distributed over the sample according to the field pattern, and $\epsilon_T = \tanh(\Delta E/2kT)$ is the thermal electron spin polarization, where ΔE denotes the electronic Zeeman splitting.

The evolution of p_i during a sweep of the magnetic field with a superimposed modulation is calculated numerically and the simulated ESR signal in phase with modulation from the direct ESR transition w_1 is shown in Fig. 7. The simulated absorption line shape is very close to the measured one and most of the unusual effects such as the broadening of the hysteresis structure and the signal enhancement by DNP at high microwave power are also correctly reproduced. However, the poor simulation of the dispersion part indicates the limits of our simplified model. Therefore, the parameters used in the simulation ($T_{2e}=10^{-8}$ s, $T_{1e}=10^{-4}$ s, $T_{1n}=1$ s, $\alpha^2=1.5\times10^{-13}$, and $\Delta B=50$ mT) should be considered only as rough estimations (orders of magnitude) of the exact values.

On the other hand, the field position of the curves is very sensitive to the *g* factor assumed for the simulation (here g = -0.4163). However, we prefer to determine the *g* factor from the raw data as described in Sec. V B in order to illustrate that the quantitative simulation is not necessary to determine this value rather accurately. Higher precision, obtained by the numerical simulation, could be meaningless because of possible systematic errors in the many-parameter fitting procedure.

In the frame of an extended model including nuclear spin diffusion and electron hopping, the observed line shapes could possibly be reproduced exactly. Our measurements could then be used to determine the numerous parameters involved precisely, provided some of them are measured independently by different experiments.

VI. DISCUSSION

Regarding the estimated electron spin relaxation times, one has to keep in mind that we deal with an electron system with reduced mobility. Hopping between the donor sites determines the electrical conductivity and may be responsible for the short T_{2e} time. In contrast to the high-conductivity regime, T_{1e} can be much longer than T_{2e} .

Concerning the nuclear spin relaxation time, it is interesting to note that the value estimated from our experiments $(T_{1n} \approx 1 \text{ s at } T = 1.4 \text{ K})$ is consistent with optical measurements involving nuclei close to a shallow donor site in GaAs (Ref. 17) (80 ms in the center of the donor wave function), but it is remarkably short compared to values reported for nuclei close to a 2DEG in GaAs (Ref. 18) or for those interacting with strongly delocalized electrons in other bulk materials.¹⁹ In these latter cases, relaxation times easily exceed tens of minutes. The effects of localization of the electron are to create an inhomogeneous nuclear polarization, with high relaxation rates in the vicinity of an electron, and to make cross relaxation of electron and nuclear spins (Korringa relaxation) more efficient due to a longer correlation time.^{15,17} On the other hand, if the electrons and the nuclear spins are almost completely isolated, T_{1n} is very long, as reported for Si:P.²⁰ A certain electron mobility, corresponding to an energy bandwidth of the order of the electronic Zeeman energy, is necessary for efficient Korringa relaxation.¹⁵ In any case, the fact that the nuclear spin relaxation rates vary by orders of magnitude depending on whether the electronic states involved are localized or extended may be essential when studying the nuclear spin relaxation in the presence of a two-dimensional electron gas in the regime of the quantum Hall effect.^{18,21,22}

VII. CONCLUSION

In summary, we have observed electron spin resonance of electrons localized on shallow donors in GaAs, using an inhouse 40-60 GHz spectrometer. The unusual shape of the ESR response is well understood in terms of dynamic nuclear polarization as confirmed by the combined ESR/NMR experiments and reproduced in model calculations. The electron *g* factor has been determined with good precision, which can be useful to increase the accuracy of band-structure and effective-mass-approximation models of GaAs. The experiments indicate that spin-dependent properties in

the GaAs matrix strongly depend on the degree of the local-

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ACKNOWLEDGMENTS

We are grateful to G. Denninger (Stuttgart) for very fruitful discussions. The LPE grown sample has been provided by E. Bauser (Stuttgart). The work of M. S. has been supported by "DAAD-Doktorandenstipendium aus Mitteln des zweiten Hochschulsonderprogramms."

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