Optically enhanced high-field NMR of GaAs

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The ^{69}Ga NMR signals of differently doped GaAs samples were directly observed under optical pumping conditions in high magnetic field (4.2 T). This technique greatly enhances the sensitivity of the NMR signals and allowed us to study the effect of optical pumping on the nuclear polarization under conditions very different from optically detected NMR. The strongest NMR signals were observed when pumping with a light energy below the band gap, suggesting the involvement of impurities and defects for the most efficient polarization transfer. Inversion of the helicity of the pumping light at the lowest temperatures results in a phase inversion of the NMR signal of a *bulk*-GaAs and a Si-doped sample, but no phase inversion was observed for a Be-doped sample.

I. INTRODUCTION

In the first optical pumping study of a semiconductor, Lampel[1] used direct nuclear magnetic resonance (NMR) detection of the ^{29}Si resonance in silicon to probe the optical orientation of electron spins via dynamic nuclear polarization[2] in a small magnetic field (1 G) achieving an enhancement factor of 10^4 over thermal equilibrium. Since then, optical pumping has been widely applied to probe nuclear spins in semiconductors[3] by optical detection of nuclear magnetic resonance (ODNMR).[4] In ODNMR, the dynamically enhanced nuclear polarization is detected indirectly under NMR conditions from the decrease of the degree of circular polarization of the recombination radiation induced by the nuclear field. Thus ODNMR presents the very high sensitivity typical of optical experiments and allows probing a very small number of nuclear spins, inaccessible by conventional NMR.

Although directly detected NMR of the optically enhanced nuclear polarization is inherently less sensitive, it presents some advantage over ODNMR, which makes this approach very promising for the study of semiconductor nanostructures. While ODNMR probes the carrier relaxation by radiative transitions, direct NMR detection does not require radiative recombination. Moreover, ODNMR is only sensitive to the influence of the nuclei located close to shallow donors or impurities where the hyperfine interaction is sufficiently strong, whereas direct NMR detection also probes nuclear spins far away from the recombination centers.[5] Furthermore, the high development of multiple pulse, multidimensional and double resonance techniques[6] allows the selective study of specific interactions between nuclei that are not accessible by optical detection methods.

In this work, we present a systematic study of the dependence of phase and magnitude of the NMR signal on the helicity and energy of the pumping light at different temperatures for differently doped GaAs samples. This study allows us to understand which electronic states are involved in the mechanism of polarization transfer. A comparison with the experiment of Barrett *et al.*[7] in quantum wells yields substantial differences, which probably can be attributed to the effects of the electron confinement in their sample. Several unexpected results highlight the different underlying physics with respect to ODNMR.

II. PRINCIPLES

The irradiation of a GaAs crystal in an external magnetic field with circularly polarized light creates spin-polarized photoelectrons in the conduction band. This nonequilibrium polarization can be transferred to the nuclear spin system via the hyperfine interaction and results in a dramatic increase of the nuclear polarization. In ODNMR, the nuclear field experienced by the electrons is tilted by applying a rotating field $\mathbf{B}_1$ near the resonance of the nuclear species. The subsequent depolarization of the electronic spin system due to the Hanle effect is detected from the decrease of the degree of circular polarization of the luminescent light. As the relevant hyperfine interaction involves the photoelectrons trapped on shallow donors, the nuclei detectable by ODNMR are only those situated close to those donors.[5] This is not a limiting factor when the nuclear polarization is observed directly, as in our experiment. In fact, in this case also the nuclei far from the impurities that are polarized via nuclear spin diffusion processes contribute to the detected signal. The entire process can be summarized in three steps: (i) The polarized light creates polarized photoelectrons in conduction band and shallow donor or impurity states; (ii) the nuclei in a distance of the order of one Bohr radius (~100 Å) (Ref. 5) to the donor are polarized by the contact with the nonequilibrium electron spin system: and (iii) spin diffusion causes polarization of the nuclei far from the donors. Due to the very different time scales, these processes can be analyzed independently. In the following, these three steps will be discussed in more detail.

(i) Polarization of the electrons. Irradiation of GaAs with $\sigma^+(\sigma^-)$ circularly polarized light induces electronic transitions from the valence band to the conduction band with a

change of the magnetic quantum number of $\Delta M_J = +1$ (-1), where M_J is the projection of the total angular momentum J. In a cubic direct-gap semiconductor such as GaAs, the transitions at the center of the Brillouin zone ($k=0$) from the sublevels of the valence band, split by the spin-orbit coupling, and the different M_J states obey different probabilities, so that the maximum net polarization of the electronic spins P_0 is either -0.5 (σ^+) or $+0.5$ (σ^-):

$$P_0 = \frac{n_+ - n_-}{n_+ + n_-}, \tag{1}$$

where $n_+(n_-)$ are the populations of the electronic states with spin up (down) with respect to the external magnetic field $\mathbf{B}_0$, which is parallel to the direction of light propagation. Taking into account the loss of spin polarization due to the recombination photoelectrons and to spin relaxation processes, the actual steady-state polarization P can be written as

$$P = P_0 \frac{\tau_s}{\tau + \tau_s} = P_0 \frac{1}{1 + \tau/\tau_s}, \tag{2}$$

where τ is the electron lifetime and τ_s is the spin relaxation time in the conduction band.[8] Thus the electronic polarization depends on the ratio of τ/τ_s, which can vary in a wide range depending on temperature and concentration of impurities, defects, and dopants.[9]

The preceding description is correct for free electrons in the conduction band, while it has been shown[5] that the relevant electrons for the polarization transfer to the nuclei are the trapped ones. Evidence of efficient averaging between free and localized electronic states has been presented in Ref. 10, where the depolarization of the electrons due to the nuclear fields has been detected by ODNMR in both localized and free-electron luminescence lines. The characteristic time of this exchange is $\sim 10^{-11}$ s, about two orders of magnitude shorter than the electron lifetime. Thus, from the point of view of the hyperfine interaction, the various electronic states are ''seen'' by the nuclei as a single spin state.[10] The effective g factor g^* results from the g factors of the different electronic states. However, it has been shown by ODNMR that in GaAs the effective g factor is negative and close to the g^* of the conduction band.[10] The only states that could affect g^* are in fact the excitonic states, but due to the short excitonic lifetime, the concentration of excitons is much smaller than the concentration of electrons.[10]

(ii) Electron-nuclear polarization transfer. Due to the hyperfine interaction term in the electron–nuclear-spin interaction Hamiltonian, the polarization of the electronic spins is transferred to the nuclei. Under steady-state conditions and considering that $|\gamma_n B_0 h/2\pi| \ll |g \mu_B B_0|$, the nuclear polarization P_N, defined as $(N_+ - N_-)/(N_+ + N_-)$, can be written as

$$P_N = \frac{P_{\mathrm{eq}} - P}{1 - P_{\mathrm{eq}} P} \frac{T_1}{T_1 + T_{1e}}, \tag{3}$$

where P_{eq} is the spin polarization of the electrons in the magnetic field $\mathbf{B}_0$ in equilibrium with the lattice temperature T: $P_{\mathrm{eq}} = \tanh(g \mu_B B_0/2kT)$. Note that P_{eq} depends on the sign of the electron g factor. $P_{\mathrm{eq}} > 0$ if $g > 0$ (for a free electron, $g \approx 2$) and $P_{\mathrm{eq}} < 0$ if $g < 0$ [in the GaAs conduction band $g^* = -0.44$ (Ref. 11)]. P is defined as in Eq. (2). T_{1e} is the nuclear relaxation time due to the interaction with the polarized electrons and T_1 is the nuclear relaxation time due to other possible mechanisms. This applies to the nuclei in the vicinity of the trapped electrons (radius of 100 Å) responsible for the polarization transfer, according to the picture of Paget.[5] The steady state of the nuclear polarization is reached in $\sim T_{1e}$, where T_{1e} has been estimated ~ 0.1 s.[9] The electron steady-state polarization is reached in a time that is comparable to τ_s and τ, i.e., several orders of magnitude faster.

When pumping with linearly polarized light, $P_0 = 0$ and the enhancement of the nuclear polarization is due to the Overhauser effect. By comparing the resulting sign of P_N with the sign of the polarization of the nuclei without any optical irradiation, it is possible to determine the sign of the g factor (with respect to the nuclear γ).

According to Eq. (3), pumping with σ^+ or σ^- light results in a nuclear polarization of opposite sign since $|P| > |P_{\mathrm{eq}}|$. No inversion of the nuclear polarization upon switching from σ^+ to σ^- light occurs when $|P| < |P_{\mathrm{eq}}|$, e.g., when strong electron-spin relaxation mechanisms are involved.

(iii) Nuclear-spin diffusion. The nuclear polarization in regions far from the shallow traps ($r > 140$ Å, according to Paget[5]) is determined by nuclear-spin diffusion. The time scale t for the nuclear polarization to diffuse a distance r is given by $t = r^2/D$, where D is the diffusion coefficient. In the case of ^{75}As in GaAs, D is $\sim 10^{-13}$ cm^2 s^{-1}, which gives ~ 10 s as a typical time to diffuse 100 Å away from the trap. Pumping for a sufficiently long time can result in a polarization of the whole crystal, compatible with the nuclear T_1, which is usually on the order of 10^3 s for a pure GaAs crystal at liquid-helium temperature.

III. EXPERIMENT

As a light source for near infrared laser light, a titanium sapphire laser (Schwartz) pumped by an argon-ion laser (Coherent) was used. The propagation vector of the laser light was aligned in parallel with the field of the superconducting magnet ($\mathbf{B}_0 = 4.2$ T) used for NMR detection. Insertion of a quarter-wave plate in the beam path allowed the variation of the helicity of the pumping light. For the entire wavelength range studied, the degree of circular polarization was higher than 90%. All laser powers were measured directly at the output of the titanium sapphire laser. After passing the quarter-wave plate, about 75% of the original power is retained.

For temperature control, the NMR probe with the sample is kept in a cryostat that is located in the bore of the superconducting magnet. An optical window at the bottom of the cryostat allows sample irradiation. Two different types of cryostats were used: a homebuilt bath cryostat and a dynamic flow cryostat (Oxford Instruments). Most experiments were performed in superfluid helium. A pressure gauge connected to the He reservoir allowed controlling the temperature of the He. Moreover, the temperature and the level of the liquid He were monitored by carbon resistors attached to the NMR probe.

The NMR probes used for the two different types of cryostats were of the same transmission line design. The tuning and matching components were kept at room temperature. The sample was mounted with a small amount of vacuum grease on a sample holder fixed at the end of the transmission line. The solenoid NMR coil was made from thin wire looped in four turns around sample and support, thus blocking only a small fraction of the sample surface from the incident light.

The distance between the optical window (in the case of the bath cryostat, the inner optical window) and the sample was about 8 cm. Due to this spacing, operation at 4.2 K was impossible since the scattering of the laser light prohibited sample irradiation. Thus all experiments in the bath cryostat were performed at about 1.7–2 K, whereas the dynamic flow cryostat also allowed operation with gaseous He as a cooling medium. In this case, temperature control was maintained with an Oxford ITC 503 unit.

Optically pumped ^{69}Ga NMR spectra at a Larmor frequency of 42.9 MHz were obtained by presaturating any equilibrium polarization with a train of 90° pulses spaced by a 50-ms delay. A shutter, inserted in the beam path and controlled by the NMR spectrometer, was kept closed during presaturation. The shutter was then opened for the irradiation period τ_L and closed again for a preacquisition delay of 500 ms and subsequent data acquisition. The free induction decay was stimulated either by a single 90° pulse of a duration of typically 9 μs or, in some cases, by a solid echo sequence consisting of two 90° pulses spaced by a delay of typically 50 μs to prevent baseline distortions due to long dead times of the probe. All data present single scan spectra; the signal-to-noise ratio was sufficiently high without signal averaging. For strong signals, the absence of a phase cycle caused the occurrence of small quadrature images in the spectra. In experiments with varying laser power or pumping time, sometimes the signal had to be attenuated to avoid saturation of the preamplifier.

Three different samples were studied. The first sample, hereafter referred to as "*bulk*-GaAs," was high resistivity GaAs used as a substrate for molecular-beam epitaxy (MBE). The other two samples consisted of 1-μm-thick GaAs layers grown by MBE on bulk-GaAs. The MBE layers of these two samples were doped with Si (1.2×10^{18} cm^{-3}) and Be (1.7×10^{18} cm^{-3}), respectively. All three samples were 0.5 mm thick and of comparable area (approximately 25 mm^2). The spot size of the laser on the sample was 4 mm^2.

IV. RESULTS AND DISCUSSION

In Fig. 1 the ^{69}Ga NMR spectra of *bulk*-GaAs are shown under different experimental conditions. Spectrum (a) was acquired at room temperature. The linewidth of the resonance (full width at half maximum) is 2 kHz. The absence of any quadrupolar satellite peaks in the spectrum is a good indication that no strain was imposed on the crystal when mounting it onto the sample holder. Spectrum (b) shows the signal that was obtained under optical pumping conditions with σ^+ light using the timing sequence described in Sec. III. The irradiation time τ_L was 20 s. Spectrum (c) was recorded under the same conditions, but with the shutter closed during the irradiation and acquisition time. Changing the helicity of the pumping light results in an inversion of the NMR signal as shown in Fig. 1(e).

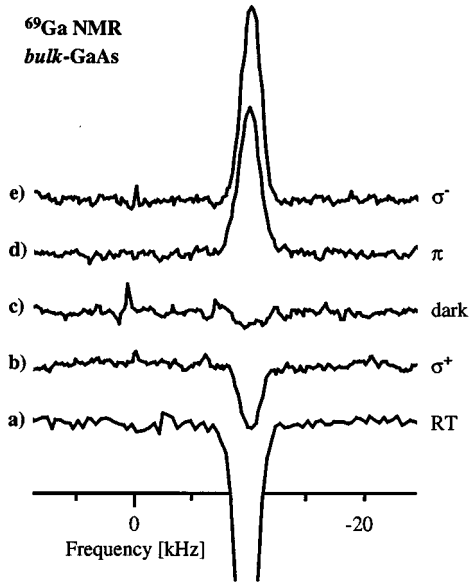

FIG. 1. ^{69}Ga NMR spectra of the *bulk*-GaAs under different experimental conditions. (a) Room-temperature spectrum, eight scans. The signal phase has been arbitrarily set to $-180°$. For all other spectra, the same phase parameters were applied. (b) Optically enhanced signal with right-handed helicity at 1.5 K. The laser power is 930 mW; light energy 1.49 eV, and $\tau_L=20$ s. (c) Same parameters as (b), but with the shutter closed during τ_L. (d) Same parameters as (b), but with linearly polarized light. (e) Same parameters as (b), but with left-handed helicity of the pumping light.

The intensity of the signal acquired at room temperature (a) is much stronger than that of the optically enhanced signals (b), (d), and (e). Whereas in the Boltzmann equilibrium case (a) nuclear spins from the whole crystal contribute to the NMR signal, only the small fraction of the sample volume exposed to the irradiation light is probed by the optical enhancement technique.

The linewidth of the signal obtained under optical pumping conditions (1.8 kHz) did not change with respect to the conventional signal recorded at the same temperature. As described in Sec. III, data acquisition was performed after a 500-ms preacquisition delay in the dark. This delay is much longer than the electron lifetime and spin relaxation time. Saturation with linearly polarized light results in an equal population of the electronic spin orientations, so that no net polarization is achieved. In this case, no signal can be observed under ODNMR conditions.[10] In our case, however, a strong ^{69}Ga NMR signal was obtained [Fig. 1(d)] since the saturation of the electronic transition results at this low temperature in a strong Overhauser enhancement of the nuclear resonance.[12,13] The 180° phase shift of this signal with respect to the Boltzmann equilibrium NMR signal (a) is due to the negative-g factor in GaAs. Thus the NMR signal phase allows the assignment of the absolute sense of circular polarization to a certain setting of our quarter-wave plate.

Figure 2 shows the dependence of the integrated ^{69}Ga NMR signal intensity of the energy of the pumping light at constant power for the three different samples. The onset of

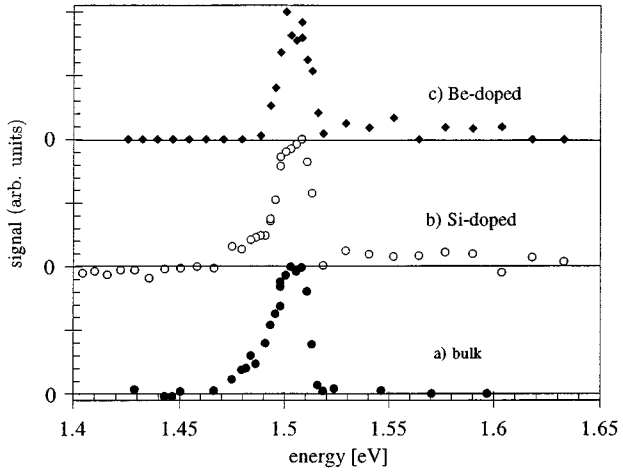

FIG. 2. Energy dependence of the integrated ^{69}Ga NMR signal intensity for the different samples at 1.5 K: (a) *bulk*-GaAs, laser power 500 mW, $\tau_L = 5$ s; (b) Si-doped GaAs, laser power 200 mW, $\tau_L = 20$ s; (c) Be-doped GaAs, laser power 200 mW, $\tau_L = 10$ s.

NMR signal observation occurs in the range 1.47–1.48 eV, peaking at about 1.50 eV. At 1.7 K, E_g of GaAs is 1.52 eV. A band-gap energy of 1.50 eV corresponds to a sample temperature of about 90 K. It is not likely that such a significant local heating was achieved. Moreover, the heating of the sample should increase with the irradiation power. For the bulk sample at 1.5 K and different irradiation powers (200 mW with $\tau_L = 60$ s, 500 mW with $\tau_L = 5$ s, and 930 mW with $\tau_L = 20$ s, the maximal NMR signal was always observed for the same excitation energy of 1.50 eV. This shows that, unlike the situation in low field ODNMR, direct population of the shallow states below the gap by optical pumping is effective for the polarization transfer to the nuclei. The increased penetration depth of the laser light below the band gap may favor polarization transfer to a larger number of nuclei from shallow states. These states can be due to shallow traps or excitons. However, due to their short lifetime, no experimental evidence has been presented so far of nuclear polarization transfer from excitonic states.[14]

The most striking feature in Fig. 2 is the narrow energy range (approximately 0.02 eV) in which a NMR signal was observed. According to theory,[3] a nuclear polarization enhancement should also be observable above the gap in the range $E_g \leqslant E \leqslant E_g + E_{SO}$,[3] where E_{SO} is the spin-orbit coupling (for GaAs, $E_{SO} = 0.34$ eV). The tuning range of our laser did not allow us to excite electrons from the Γ_7 subband, so that for $E > E_g$ transitions from Γ_8 at $k \neq 0$ are induced, creating hot electrons in the conduction band. Thermalization of hot electrons to the bottom of the conduction band in pure and *n*-type crystals is usually accompanied by electron-spin relaxation due to the D'yakonov-Perel mechanism.[15,16] Therefore, we expect a decrease in the nuclear polarization for $E > E_g$, which is in agreement with the experimental results [Figs. 2(a) and 2(b)]. For *p*-type crystals, however, the relaxation of hot electrons occurs under retention of the polarization,[8] and an almost-energy-independent electron polarization is found in the range $E_g \leqslant E \leqslant E_g + E_{SO}$. This should also be reflected in the nuclear polarization. Figure 2(c) shows the result for the *p*-type GaAs sample. For energies larger than 1.52 eV, only a weak

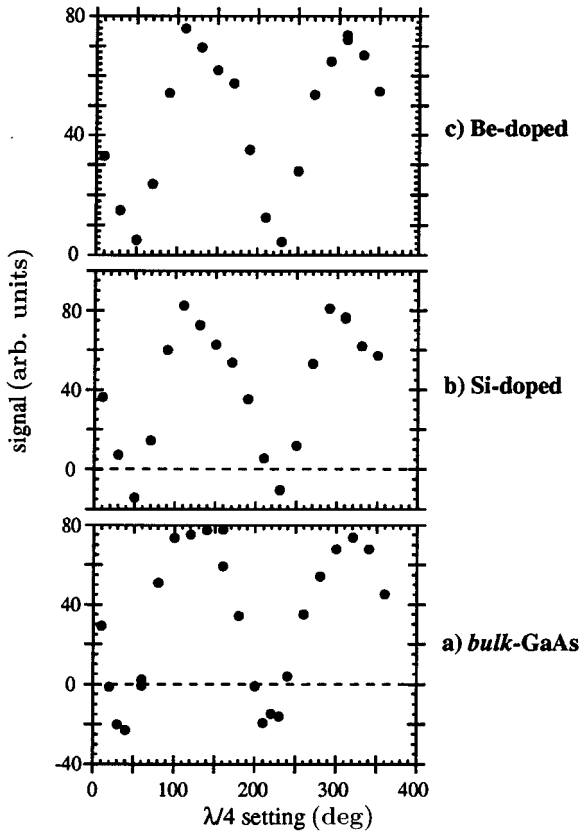

FIG. 3. Dependence of the integrated ^{69}Ga NMR signal intensity on the helicity of the pumping light, expressed in the setting of the quarter-wave plate. σ^+ corresponds to 130° and 310°, σ^- to 50° and 230°, and π to 5°, 95°, 185°, and 275°. $T = 1.5$ K. For *bulk*-GaAs and Si-doped GaAs, the zero signal intensity is marked by the dashed line to underline the regions of phase inversion upon switching from σ^+ to σ^- light. (a) *bulk*-GaAs, laser power 930 mW, light energy 1.49 eV, $\tau_L = 10$ s. (b) Si-doped GaAs, laser power 200 mW, light energy 1.49 eV, $\tau_L = 20$ s. (c) Be-doped GaAs, laser power 200 mW, light energy 1.49 eV, $\tau_L = 10$ s.

NMR signal was observed. This result seems to reveal an effective hot-electron-spin relaxation mechanism in this high magnetic field.

No frequency shift of the NMR signal is observed with respect to the position in Figs. 1(b) and 1(e). In ODNMR, a shift of the resonance line is usually observed, since only nuclei are detected within one Bohr radius of the shallow donor, where a high density of polarized electrons are trapped. In a GaAs/$Al_xGa_{1-x}As$ quantum well sample,[7] a Knight shift was observed with direct NMR detection. In this case, the high density of conduction electrons is probably determined by the quantum confinement of the photoexcited electrons. In our case, the NMR signal arises most likely from nuclei located far away from the traps and the nuclear polarization is created by spin diffusion rather than by direct hyperfine interaction. Moreover, a nuclear polarization induced by contact with conduction electrons is excluded, since the light energy was below the band gap.

In Fig. 3, the dependence of the intensity and phase of the NMR signal on the polarization of the pumping light is

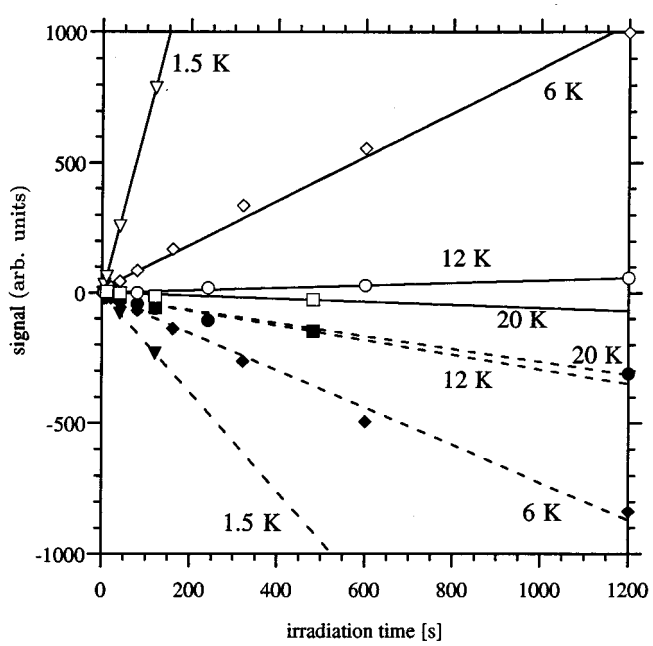

FIG. 4. Dependence of the integrated ^{69}Ga NMR signal intensity on the pumping time τ_L at 1.5 K with a laser power of 200 mW and a light energy of 1.50 eV for *bulk* GaAs at different temperatures. As a guide to the eye, the data points were connected with lines resulting from a linear fit, although at higher temperatures a saturation level is reached. This is especially obvious for the data at 6 K. Empty symbols and full lines represent data taken with σ^- irradiation, whereas full symbols and dashed lines represent those with σ^+ light. For each pair of data at one temperature, the same type of symbol was used.

shown for the different samples. The curves show a different offset from 0, which is so strong in the case of the Be-doped sample that no inversion of the NMR signal is observed. In other words, the ratios of the efficiency of nuclear polarization transfer for σ^+ and σ^- light is strongly dependent on the dopants in the sample. In Figs. 3(a)–3(c), the respective integral of the dark signal has been already subtracted, so that nuclear T_1 effects of the different samples do not contribute to the effect shown in Fig. 3.

As shown in Eqs. (2) and (3), the absence of inversion of the nuclear polarization obtained by irradiating with σ^+ and σ^- light can be caused by a large value of the ratio τ/τ_s. Thus the experimental results are consistent with a more efficient electron-spin relaxation in the Be-doped sample, probably due to exchange with holes. However, other mechanisms may play a role, since the recombination process can depend on the spin of the recombining electrons. Such effects have been observed with ODNMR in heavily-manganese-doped GaAs.[17] Because of the high magnetic field applied in these experiments, polarization of the valence holes and acceptors can occur, resulting in different recombination rates for the photoelectrons with spin up or down. This effect may be important in the Be-doped GaAs sample.

The effect of the different pumping efficiencies in terms of nuclear polarization P_N was more carefully investigated as a function of the irradiation time for different temperatures and laser powers. Figure 4 shows the results obtained for the *bulk*-GaAs at constant power and variable temperature for σ^+ and σ^- irradiation. The dark signal has been already subtracted for the individual data points. With decreasing temperature, the steeper slope of the lines indicates an increase in the pumping efficiency due to the longer lifetime and spin relaxation time of the photoexcited electrons. For temperatures in the range 1.5 K $\leqslant T \leqslant$ 12 K, an inversion of the phase of the NMR signal with opposite helicity of the pumping light is observable. At $T=20$ K, the signal phase for σ^+ and σ^- irradiation is the same as for the conventional Boltzmann signal. This fact seems to be contradictory to the observation that the electronic g factor is negative. Such a behavior can be accounted for when considering that the photoexcited electrons also cause a shortening of the nuclear-spin lattice relaxation time. Thus the signal observed at higher temperatures is primarily the Boltzmann magnetization built up during the irradiation time.

V. CONCLUSION

In this work, we have revealed several unexpected effects in the high field optical pumping of GaAs coupled with direct NMR detection. Due to the longer lifetime and relaxation time of the electrons, we observe, according to the expectations, the strongest NMR signal at the lowest temperatures applied (1.5 K). However, at this temperature for all samples (n-type, p-type, and undoped GaAs), the strongest NMR signal corresponds to a light energy of 1.5 eV, while the band gap at this temperature is 1.52 eV. A possible explanation is heating of the sample upon irradiation, but in this case a shift of the maximum NMR signal to lower energies is expected with increasing laser power, which is not in agreement with the experimental results. More likely, states below the band gap are involved in the polarization transfer process. These states may be favored because of the increased penetration depth of the light. An independent verification is desirable by detecting the degree of circular polarization of the luminescence light simultaneously with the NMR signal. No shift of the NMR signal with respect to an acquisition in the dark is observed. Such a shift is expected if the nuclei contributing to the NMR signal are in the proximity of a paramagnetic center or interacting with conduction electrons. In our case, no conduction electrons were generated as the excitation energy was smaller than the band gap. However, there is a sufficient number of electronic states present to generate a large nuclear polarization. These states may be trapped on impurities and defects. Therefore, we conclude that the NMR signal is due to nuclei that were polarized by spin diffusion and are not located in the proximity of dopants or defects.

Upon changing the helicity of the pumping light, NMR signal inversion was observed for the *bulk* and the *n*-type GaAs, but not for the *p*-type sample. This is consistent with a faster electron-spin relaxation mechanism in the *p*-type sample, although other mechanisms, such as a spin-dependent electron recombination, could also be relevant.

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