Electrical detection of cross relaxation between electron spins of phosphorus and oxygen-vacancy centers in silicon

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We report on the electrical detection of cross relaxation (EDCR) processes in phosphorus-doped γ-irradiated silicon, where the dipolar-coupled electron spins of phosphorus and oxygen-vacancy complex (Si-SL1 center) undergo spin flip-flop transitions at specific magnetic field values for which the Zeeman splitting of the two centers become equal. Such cross relaxation signals are observed as the change in the sample photoconductivity at theoretically predicted magnetic fields *without* application of resonance frequency. This EDCR is a very simple and sensitive method for detecting paramagnetic centers in semiconductors.

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I. INTRODUCTION

In the presence of external magnetic fields, the spin population among magnetic sublevels approaches the Boltzmann distribution in a time frame known as spin-lattice relaxation time (T_1).[1,2] When two different kinds of spins coexist in solid, they can achieve identical spin temperature through energy conserving flip-flop transitions, provided that their Zeeman energies are made nearly equal by tuning the magnetic field strength.[3–5] This phenomenon is known as cross relaxation and occurs if the exchange of energy between the two different spins is significantly faster than the exchange with the lattice, i.e., $\tau_{CR} < T_1$, where τ_{CR} is the cross relaxation time.[6,7] In the past, optical detection of cross relaxation (ODCR) has been studied in solids, where the change in the luminescence intensity from one of two different dipolar coupled paramagnetic centers is monitored as their Zeeman splittings are brought into resonance by appropriate tuning of the magnetic field.[8,9] However, the optical method can be used only if recombination through one or both the paramagnetic centers are radiative.

The present work reports electrical detection of cross relaxation (EDCR) between two different paramagnetic centers in silicon. So far, an electrical method for detecting magnetic resonance, widely known as electrically detected magnetic resonance (EDMR), has been demonstrated for a variety of condensed-matter systems. EDMR detects changes in photoconductivity via spin-dependent recombination (SDR)[10–13] or spin-dependent scattering[14,15] when target paramagnetic centers are brought into resonance with externally applied radio or microwave fields. The present work on the electrical method for detecting cross relaxation probes the change in photoconductivity when two different spin systems are brought into resonance by tuning the magnetic field. Unlike the case of electron paramagnetic resonance (EPR) and EDMR spectroscopy, EDCR does not require external irradiation to induce transitions between the magnetic sublevels because two different centers that are coupled by magnetic dipolar interactions undergo energy-conserving flip-flop transitions. Therefore, EDCR measurement is as simple as monitoring photoconductivity under a scanning magnetic field and applicable for detecting both radiative and nonradiative centers.

The present study focuses on the cross relaxation between electron spins of phosphorus and oxygen-vacancy centers (A-centers) in a γ-ray irradiated Czochralski (CZ)-grown, phosphorus (P)-doped silicon single crystal. A-centers are known to be the dominant defects created by irradiation of oxygen-rich CZ silicon.[16,17] A-centers can be easily transformed by band-gap illumination into excited triplet states (electron spin $S= 1$) that lead to well-known Si-SL1 EPR spectra.[18] Thus, under illumination the sample contains predominantly two kinds of paramagnetic centers: phosphorus ($S = 1/2$ and ^{31}P nuclear spin $I = 1/2$) and Si-SL1 centers ($S = 1$). Figure 1 shows the Zeeman levels of SL1 and phosphorus as a function of the applied magnetic field. Cross relaxations are expected when Zeeman splittings of phosphorus and SL1 centers are made equal by tuning the magnetic field.

The structure and spin Hamiltonian parameters of the triplet SL1 centers are well established.[18] The SL1 center has orthorhombic symmetry represented by g and D tensors. They orient along six different $\langle 110\rangle$ crystal axes of the silicon lattice. When the magnetic field B is rotated in one of $\{110\}$ planes, the angle between the magnetic field and one of the six groups of SL1 centers [represented by SL1^0 in Fig. 1(a)] is varied from 0° ($B\|\langle 110\rangle$) to 90° ($B \perp \langle 110\rangle$). For the second group [represented by SL1^{90} in Fig. 1(b)], the angle is always equal to 90°. The remaining four groups make intermediate angles with the magnetic field. Phosphorus in silicon, on the other hand, is a much simpler system of electron spin ($S = 1/2$) coupled to a nuclear spin ($I = 1/2$) via an isotropic hyperfine interaction $A/2\pi \approx 117.5$ MHz. Figure 1(c) shows the Zeeman splitting between magnetic sublevels of phosphorus as a function of the magnetic field.[19]

The two centers interacting with each other through the long-range dipole-dipole interaction which accounts for the flip-flop transition is given by[6]

$$\mathcal{H}_{DD} = \frac{\gamma_P \gamma_{SL1} \hbar^2}{4r^3}(S_-^P S_+^{SL1})(1 - 3\cos^2\Theta), \qquad (1)$$

where γ_P and γ_{SL1} are the gyromagnetic ratios of phosphorus and SL1 centers, respectively, r is the distance between the two centers, $S_\pm = S_x \pm i S_y$, and Θ is the angle between the magnetic field direction and vector joining the two paramagnetic

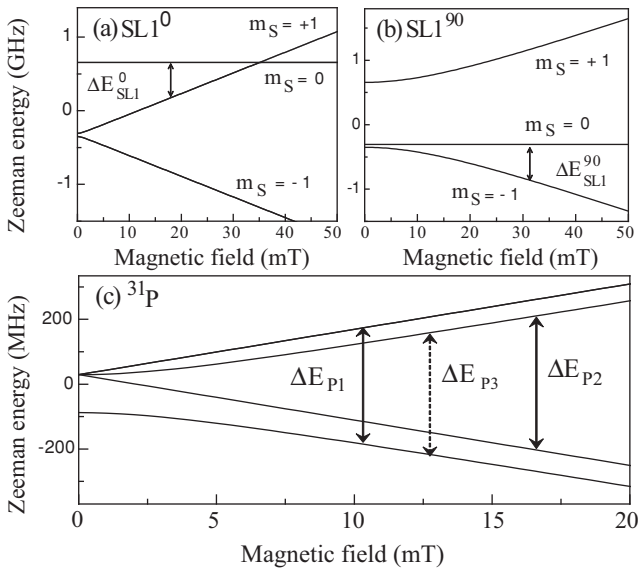

FIG. 1. (a) Zeeman energy levels of $SL1^0$ center oriented along the direction of the magnetic field, (b) $SL1^{90}$ center oriented at 90° to the applied magnetic field $B\|\langle 110\rangle$, and (c) energy levels of phosphorus (^{31}P). ΔE^0_{SL1}, ΔE^{90}_{SL1}, and $\Delta E_{P1,P2,P3}$ are the energy differences between the Zeeman levels (indicated by double-headed arrows) of SL1 centers and phosphorus, respectively. ΔE_{P3} is the forbidden transition which becomes observable only at low magnetic fields ($\leqslant 20$ mT) due to the mixing of spin states.[13]

centers. The flip-flop transition probability ($W_{CR} = 1/\tau_{CR}$) is given by[6]

$$W_{CR} = \frac{2\pi}{\hbar^2} g_{CR}(\omega_{CR}) \times |\langle m_P - 1, m_{SL1} + 1|\mathcal{H}_{DD}|m_P, m_{SL1}\rangle|^2, \quad (2)$$

where $\mathcal{H}_{DD}$ is the dipolar Hamiltonian for flip-flop transition and $g_{CR}(\omega_{CR})$ is the overlap function for cross relaxation. For Gaussian line shapes, $g_{CR}(\omega_{CR})$ is given by

$$g_{CR}(\omega_{CR}) = \frac{1}{2\pi\left(\Delta\omega_P^2 + \Delta\omega_{SL1}^2\right)^{1/2}} \times \exp\left[-\frac{(\omega_P - \omega_{SL1})^2}{2\left(\Delta\omega_P^2 + \Delta\omega_{SL1}^2\right)^{1/2}}\right], \quad (3)$$

where $\Delta\omega_P^2$ and $\Delta\omega_{SL1}^2$ are the second moments of line shapes of phosphorus and SL1, respectively. It can be seen from the above equations that the probability for cross relaxation is maximal for the magnetic field at which the Zeeman frequencies ω_P and ω_{SL1} become equal, and its probability decreases significantly as the externally applied magnetic field shifts away from the equal point.

It is clear from the above discussions that the average distance between phosphorus and SL1 centers, i.e., the concentrations of the two centers, must be appropriate to achieve strong enough dipolar coupling for induction of cross relaxation. The observation of cross relaxation signals in this study is partially due to successful control of the concentration of phosphorus and SL1 centers in the sample.

II. EXPERIMENTAL

Rectangular-shaped samples ($8 \times 4 \times 1$ mm^3) with the long edge along the $\langle 110\rangle$ crystal axis were cut from a CZ-grown n-type single crystal silicon wafer having the phosphorus concentration $\sim 10^{15}$ cm^{-3}. Ohmic contacts for electrical measurements were made by ion implantation of arsenic (dose $\approx 10^{15}$ cm^{-2} at 25 keV) followed by 30 s annealing at 950 °C and vacuum deposition of palladium and gold. After making Ohmic contacts, samples were irradiated by γ rays emitted from a ^{60}Co source at room temperature with the dose of $\sim 10^{15}$ cm^{-2} to produce $\sim 10^{13}$ cm^{-3} of A-centers throughout the sample volume. Prior to each measurement, the surface silicon dioxide was removed with dilute hydrofluoric acid (HF) solution to reduce the resonance due to surface defects.

EDCR measurements were performed at low temperatures ($T < 10$ K) in a helium bath cryostat having optical windows for illumination. A white light from a 100 W halogen lamp was focused on the sample through one of the optical windows of the cryostat. The change in sample photoconductivity under a scanning magnetic field was measured using electrical contacts. A lock-in detector tuned to the second harmonic of magnetic field modulation at a frequency 5 kHz was employed to increase the signal-to-noise ratio. The signals were recorded as second derivatives of the magnetic field. The angular dependence of the cross relaxation line position was measured by rotating the sample about the $\langle 110\rangle$ crystal axis.

The cross-relaxation spectra were also detected with the same sample using the microwave SDR photoconductivity technique.[20–22] Here an X-band (9 GHz) EPR spectrometer was used and the change in photoconductivity of the sample was detected as the variation in the cavity Q factor since the change in the concentration of photoexcited carriers leads to change in the absorption of the microwave in the EPR cavity.

III. RESULTS AND DISCUSSIONS

A. EDCR and EDMR spectra

Figure 2(a) shows the change in the photoconductivity (EDCR intensity) with a sweeping magnetic field observed without the application of an external excitation field. The difference in signs of the signal is due to the phase of the lock-in amplifier used to optimize the signal-to-noise ratio.

Figure 2(b) shows the Zeeman energy splittings, ΔE_{SL1} and ΔE_P as defined in Fig. 1, between the spin states of SL1 and phosphorus, respectively. It is apparent from Figs. 2(a) and 2(b) that the EDCR signals appear as expected at magnetic fields where $\Delta E^0_{SL1} = \Delta E_{P1}$, ΔE_{P2}, ΔE_{P3}, and $\Delta E^{90}_{SL1} = \Delta E_{P2}$. Hereafter, these cross relaxation flip-flop transitions are referred to as $\Delta E^0_{SL1} \Leftrightarrow \Delta E_{P1}$, $\Delta E^0_{SL1} \Leftrightarrow \Delta E_{P2}$, $\Delta E^0_{SL1} \Leftrightarrow \Delta E_{P3}$, and $\Delta E^{90}_{SL1} \Leftrightarrow \Delta E_{P2}$ relaxations. The signal appearing at zero magnetic field, zero-field line (ZFL), is due to the cross relaxation among degenerate levels. As reported by Bloembergen *et al.*[6] the cross relaxation can occur even within a single paramagnetic center having three or more magnetic sublevels. Both phosphorus and SL1 centers have degenerate energy levels at zero magnetic field which contribute to the zero-field signal.

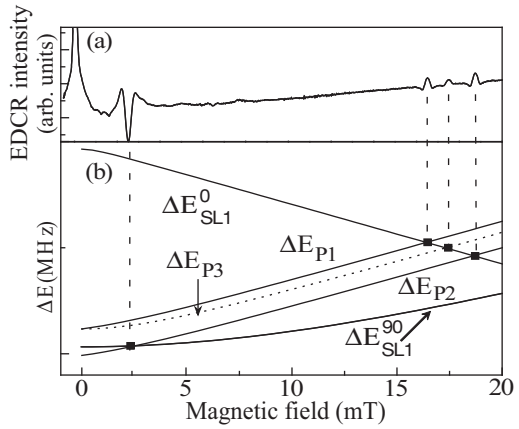

FIG. 2. (a) An EDCR spectrum showing the change in photoconductivity (EDCR intensity) with a scanning magnetic field $B \| \langle 110 \rangle$, $T = 7$ K with no resonance frequency. (b) Calculation showing the magnetic field dependence of the ΔE's as defined in Fig. 1. Solid squares (■) in (b) show the crossing points between ΔE's of SL1 center and phosphorus for $B \| \langle 110 \rangle$, where the occurrence of cross relaxations is expected.

The EDMR spectroscopy was used to validate the presence of phosphorus and SL1 centers in our sample. Figure 3 shows the EDMR spectrum from phosphorus-doped γ-irradiated silicon at 7 K with an externally applied rf field of 400 MHz. The EDMR spectrum shows the resolved ^{31}P hyperfine doublet separated by 4.2 mT and fine structures of the SL1 centers as expected.[11,22,23] These EDMR signals confirm the presence of phosphorus and SL1 centers in the sample. The signal marked

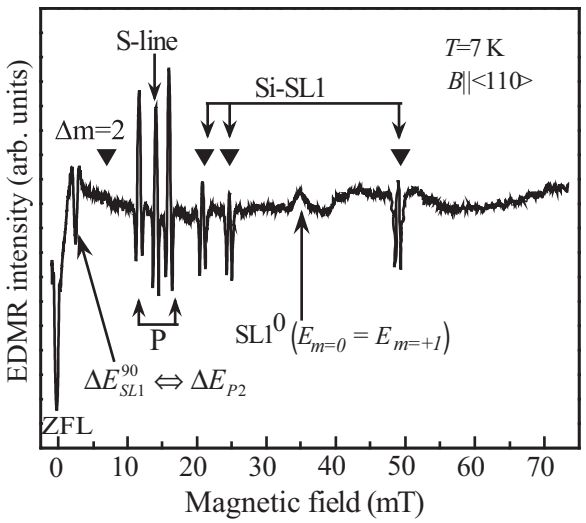

FIG. 3. An EDMR spectrum showing spin-dependent recombination signals in phosphorus-doped γ-irradiated silicon recorded with the application of a 400 MHz resonance field, $B \| \langle 110 \rangle$, and $T = 7$ K. Along with phosphorus hyperfine lines (P), signals from SL1 centers (Si-SL1) and surface centers (S-line) were observed. Solid triangles indicate calculated line positions of SL1 signals using the parameters reported in Ref. 18. The cross relaxation signal marked SL1^0($E_{m=0} = E_{m=+1}$), is independent of applied resonance frequency and appears at the anticrossing point of $m_s = 0$ and $m_s = +1$ states of SL1^0 and is observed even when the rf is turned off.

SL1^0($E_{m=0} = E_{m=+1}$) in Fig. 3 arises at the anticrossing point of $m_s = 0$ and $m_s = +1$ states in Fig. 1(a) and can be observed even without application of the resonance field. This signal is due to mutual flip-flops between the $m_s = 0$ and $m_s = +1$ states of SL1 centers that are oriented along the externally applied magnetic field.

Figure 4 shows EDMR spectra recorded with three different rf fields. The line positions of the cross relaxation signals (dotted lines) are independent of the irradiated frequency as expected, while the positions of the EDMR signals shift along with the resonance frequency. These results further substantiate our understanding of cross relaxation processes, which are independent of the applied rf field.

B. Angular dependence of EDCR line positions

In contrast to isotropic EPR and EDMR spectra of phosphorus, the SL1 centers exhibit strong anisotropy in the line positions with respect to the direction of the applied magnetic field.[18,22] Thus, the line positions of EDCR signals arising from phosphorus and SL1 centers should also be anisotropic.

Figure 5 shows the experimentally observed EDCR line positions when the sample is rotated about the $\langle 110 \rangle$ crystal axis. The angular dependence of $\Delta E^{90}_{\mathrm{SL1}} \Leftrightarrow \Delta E_{\mathrm{P2}}$ is very weak as compared to the lines labeled as I, II, III, and IV that show very strong anisotropy. Our EDCR experimental setup is currently limited to rotation of the sample up to 45°. Thus, in order to obtain the complete angular dependence in the 0° ($B \| \langle 110 \rangle$) to 90° ($B \| \langle 100 \rangle$) range, the cross relaxation line positions were obtained by the contact-free microwave photoconductivity technique using the X-band EPR spectrometer. Experimental results are shown in Fig. 6.

The angular dependence of cross relaxation line positions observed by EDCR (Fig. 5) and microwave photoconductivity [Fig. 6(b)] are essentially the same. Moreover, the

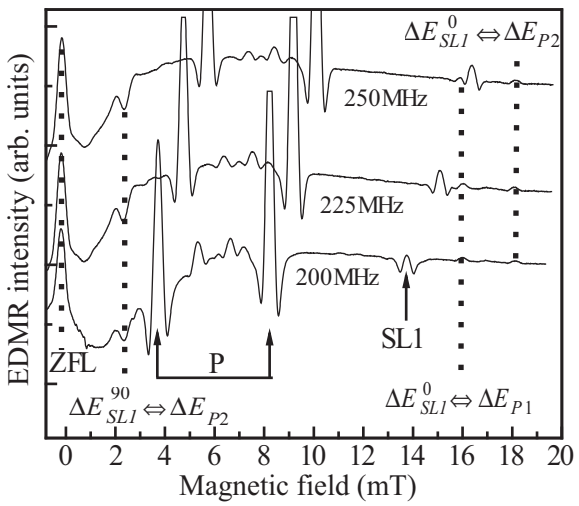

FIG. 4. EDMR spectrum with three different resonance frequencies for $B \| \langle 110 \rangle$ and $T = 7$ K. Positions of phosphorus (P) and SL1 EDMR signals (marked by solid arrows) shift with the frequency, while the positions of EDCR signals (marked by dotted lines) are independent of the frequency.

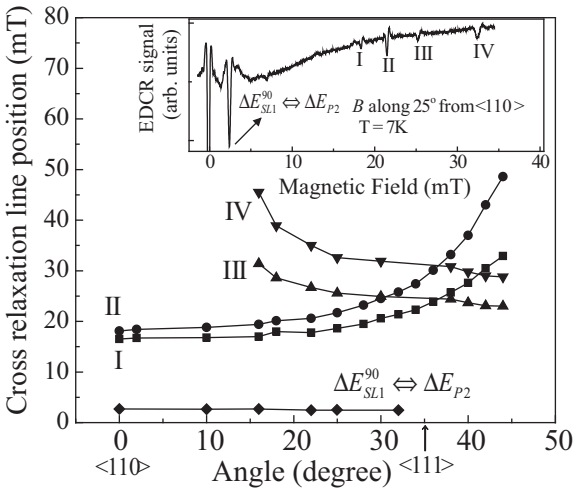

FIG. 5. Angular dependence for the line positions of EDCR signals from phosphorus-doped γ-irradiated silicon. The inset shows the EDCR spectrum when the external magnetic field (B) is applied along 25° from ⟨110⟩.

experimentally observed cross relaxation line positions show excellent agreement with theoretically calculated line positions represented by solid curves in Fig. 6(b). Calculations were performed for each of six possible orientations of SL1 centers in a silicon crystal. The $\Delta E^{90}_{\rm SL1} \Leftrightarrow \Delta E_{\rm P2}$ line has very weak angular dependence and is attributed to cross relaxation between electron spins of phosphorus and SL1 centers which are oriented perpendicular to the magnetic field throughout the rotation around the ⟨110⟩ axis. Lines I and II originate from cross relaxation between phosphorus and SL1 centers oriented in the {110} plane. The remaining four orientations of SL1 centers are responsible for lines III, IV, V, and VI.

The excellent correlation between the experimental and theoretically calculated angular dependencies confirms that the spin flip-flop process between phosphorus and spin triplet SL1 centers is responsible for the EDCR signals detected in this study.

C. Model for the observed photoconductivity change under cross relaxation

To explain the change in photoconductivity by cross relaxation, we construct a simple model for spin-dependent recombination of photoexcited carriers through the excited spin $S = 1$ states of A-centers coupled with paramagnetic P atoms by long-range dipolar interaction. P and SL1 centers separated by a distance of r can exchange energy by the spin flip-flop process, when their Zeeman splittings are made nearly equal by tuning the magnetic field strength. The energy $\Delta E_{\rm P}$ released by the electron bound to phosphorus through relaxation from $m_S = +1/2$ to $m_S = -1/2$ is absorbed by the SL1 center to induce transition from $m_S = 0$ to $m_S = +1$. These flip-flop transitions change the populations among the magnetic sublevels of SL1 centers. The steady-state populations n_+, n_0, and n_- for spin projections $m_S = +1$,

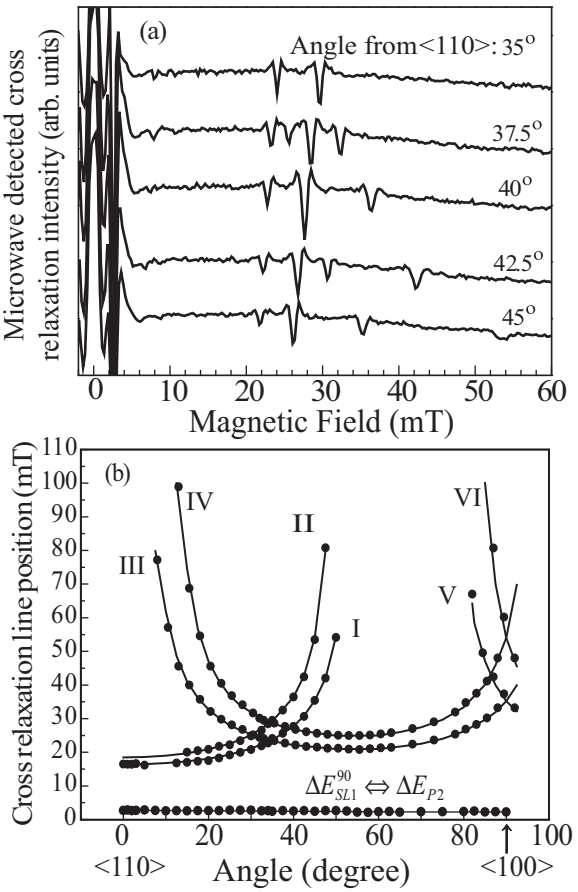

FIG. 6. (a) Cross relaxation signals detected by the microwave photoconductivity technique and (b) angular dependencies of their positions revealed by the microwave-photoconductivity technique. Dots (•) represent the experimentally obtained line positions of cross relaxation signals and curves represent the calculated positions of Zeeman energy crossing points between phosphorus and SL1 centers. Signals marked as I, II, III, IV, V, and VI originate from SL1 centers having different orientations in the crystal.

$m_S = 0$, and $m_S = -1$, respectively, of SL1 centers can be found from the following rate equations:

$$\frac{dn_+}{dt} = G_T - R_+ n_+ - (n_+ - n_0)(W + W_{\rm CR}), \quad (4a)$$

$$\frac{dn_0}{dt} = G_T - R_0 n_0 - (2n_0 - n_+ - n_-)W - (n_0 - n_+)W_{\rm CR}, \quad (4b)$$

$$\frac{dn_-}{dt} = G_T - R_- n_- - (n_- - n_0)(W + W_{\rm CR}). \quad (4c)$$

For steady-state solution $\frac{dn_+}{dt} = \frac{dn_0}{dt} = \frac{dn_-}{dt} = 0$. Here G_T is the generation rate of triplet states under illumination, which is the same for $m_S = +1$, $m_S = 0$, and $m_S = -1$ states. R_+, R_0, and R_- are the transition probabilities from $m_S = +1$, 0, and -1 to the ground singlet state, respectively. In zero-order approximation these transitions are forbidden, but taking into account the spin-orbit interaction, the probabilities R_+, R_0, and R_- are nonzero and $R_+ = R_- \neq R_0$.[24] $W_{\rm CR}$ is the probability of cross relaxation caused by other paramagnetic centers when the Zeeman splitting between these centers

coincides with the splitting between the $m_S = +1$ and $m_S = 0$ states. W is the spin-lattice relaxation probability.

The generation and recombination of photoexcited carriers can be described by the simple rate equation,

$$\frac{dn_e}{dt} = G - R_A n_e(N - N_T) - Rn_e, \tag{5}$$

where G is the generation rate of electrons by light, N is the total population of A-centers, and N_T is the population of A-centers in the excited triplet state. The second term describes the rate of formation of excited triplet states by electron recombination through A-centers.[25] The last term in Eq. (5) corresponds to the rate of electron recombination R through other defects in the sample. Using Eqs. (4) and (5), the change in photoconductivity under cross relaxation can be calculated as the difference between the steady-state populations of photoexcited electrons for $W_{CR} \neq 0$ and $W_{CR} = 0$, i.e., $\Delta n_e = n_e(W_{CR}) - n_e(0)$.

The important parameters for the detection of EDCR signals are the ratio of the cross relaxation rate to the spin-lattice relaxation rate, W_{CR}/W, and the ratio of the spin-lattice relaxation time ($T_1 = 1/W$) to the lifetime (τ_T) of the excited triplet states. Figures 7(a) and 7(b) show the dependence of $\Delta n_e/n_e(0)$ on the ratio W_{CR}/W for different values of T_1/τ_T and on the ratio T_1/τ_T at a fixed value of $W_{CR}/W = 10$. The calculations were performed for $R_+ = R_- = 10R_0$. These results have very weak dependence on the values of the parameters N, G, R_A, and R.

Figure 7 shows that the change in photoconductivity due to cross relaxation (which is proportional to Δn_e) is negative corresponding to the increase in the recombination rate at the onset of the cross relaxation process. The absolute value of Δn_e increases with the increase of the cross relaxation rate W_{CR} at fixed W. As follows from Fig. 7(b), the EDCR signals can be observed for a wide range of T_1/τ_T ratios. However, for the observation of cross relaxation signals, the following condition should be satisfied:

$$\frac{1}{\tau_{CR}} > \frac{1}{\tau_T} > \frac{1}{T_1}, \tag{6}$$

where τ_{CR} is the cross relaxation time, τ_T is the decay lifetime of the SL1 center, and T_1 is the spin relaxation time of the SL1 center. The first inequality is important for the occurrence of the cross relaxation before the SL1 center decays. The second inequality defines the criteria for the existence of nonequilibrium population distribution among the magnetic sublevels of SL1 centers. For these triplet centers, the spin-lattice relaxation time $T_1 \sim 10$ s is significantly longer than their lifetime $\sim 10^{-3}$ s,[26] thus the last inequality in Eq. (6) is satisfied. The cross relaxation time (τ_{CR}), limited by the lifetime of SL1 centers, should be $\leqslant 10^{-3}$ s. Assuming that the EPR line shape is Gaussian, it can be estimated using Eqs. (1)–(3) that a phosphorus and SL1 pair separated by $r \leqslant 20$ nm has a cross relaxation time $\tau_{CR} \leqslant 10^{-3}$ s. From the concentration of the randomly distributed paramagnetic centers in the sample, it can be estimated that the EDCR signal observed in this study has a contribution from 10^8 interacting pairs of phosphorus-SL1 centers with the separation between the phosphorus and SL1 centers distributed within the sphere of radius $r \leqslant 20$ nm.[27]

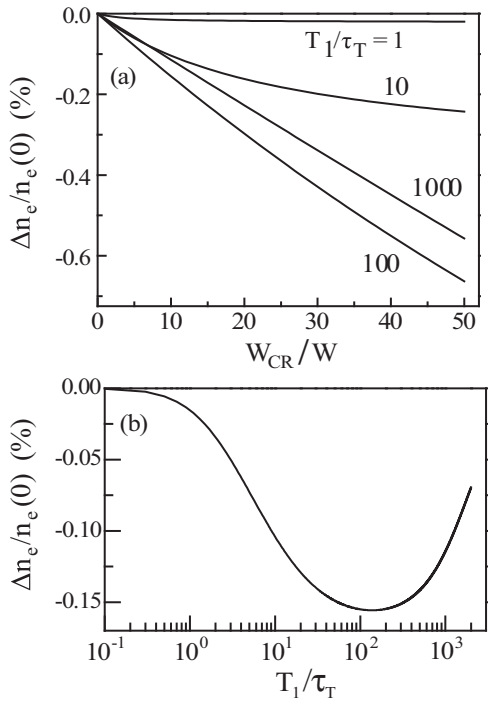

FIG. 7. (a) The relative change of $\Delta n_e/n_e(0)$ as a function of W_{CR}/W for different values of T_1/τ_T and (b) $\Delta n_e/n_e(0)$ as a function of T_1/τ_T for $W_{CR}/W = 10$.

IV. CONCLUSION

Electrical detection of cross relaxation between electron spins of phosphorus and SL1 centers in silicon has been demonstrated experimentally. The flip-flop transitions led to changes in the sample photoconductivity at theoretically expected externally applied magnetic fields. The angular dependence of the cross relaxation peak positions also agrees well with calculated values reflecting the symmetry of SL1 centers. The change in the photoconductivity originates from the difference in the lifetimes of magnetic sublevels of triplet states of SL1 centers that changes the electron-hole recombination time at the occurrence of cross relaxation. The experimental observation of EDCR signals requires the cross-relaxation rate to be higher than the decay rate of triplet centers, which in turn must exceed the spin-lattice relaxation rate. These conditions are satisfied for SL1 centers situated within the sphere of radius $r \leqslant 20$ nm from each phosphorus atom. The electrical detection of cross relaxation is a very simple and sensitive technique to investigate a variety of defects in semiconductors.

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[25]A^{0*} centers (SL1 centers) are created by the capture of photoexcited electrons (e) and holes (h) by (A^0), (A^+), and (A^-) centers according to reaction $A^0 + e \rightarrow A^- + h \rightarrow A^{0*} \overset{R_+,R_0,R_-}{\longrightarrow} A^0$ or $A^0 + h \rightarrow A^+ + e \rightarrow A^{0*} \overset{R_+,R_0,R_-}{\longrightarrow} A^0$, where the transitions $A^{0*} \longrightarrow A^0$ are spin dependent. Under illumination, part of the A-centers ($N_T = n_+ + n_0 + n_-$) are in the excited triplet state (A^{0*}). And the remaining, $N_0 = N - N_T$, are in the positive (A^+), negative (A^-), and in the neutral singlet (A^0) charge states. Here, N is the total population of A-centers in the sample.
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[27]Irradiation of the sample by γ rays emitted from a ^{60}Co source at room temperature with the dose of $\sim 10^{15}$ cm^{-2} produces $\sim 10^{13}$ cm^{-3} of A-centers throughout the sample volume. However, the above band-gap illumination intensity employed in the experiment limits the concentration of triplet excited states to $\sim 10^{11}$ cm^{-3}, i.e., $\sim 10^9$ SL1 centers in the bulk of the sample. The average separation between phosphorus atoms for the concentration of 10^{15} cm^{-2} is ~ 100 nm. If we assume one phosphorus atom interacting with one SL1 center, then there are $\sim 10^9$ phosphorus-SL1 pairs with the separation r between the centers distributed randomly between 0 and 50 nm (half the distance between a phosphorus atom). The probability that an SL1 center will be found in the sphere of radius r from the phosphorus atom is clearly $r^3/(50)^3$. Thus, we estimate that $\sim 10^8$ pairs of phosphorus-SL1 centers with the separation distributed within the sphere of radius $\leqslant 20$ nm, contributes to the EDCR signal observed in this study.