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Spin diffusion and spin–lattice relaxation in pulse spin-locking in solids containing paramagnetic impurities

Gregory B Furman and Shaul D Goren

Department of Physics, Ben-Gurion University, Be'er-Sheva, Israel

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Abstract. The spin diffusion and spin–lattice relaxation in solids containing paramagnetic impurities under the influence of a multiple-pulse spin-locking radio-frequency sequence are studied. The diffusion equation is obtained, providing the possibility of finding the time dependence of the magnetization. The spin–lattice relaxation time is calculated as a function of the correlation time and multiple-pulse field parameters.

1. Introduction

In solids containing paramagnetic impurities (PIs) the dipole–dipole interactions (DDIs) play the dominant role in the spin-relaxation processes [1, 2]. The DDI between nuclear spins I and PI spins S leads to a direct spin–lattice relaxation of the nuclear spin system and, due to the inverse-sixth-power dependence on their separation, the local nuclear magnetizations reach their equilibrium values at a faster rate near the PIs [1–3]. As a result, the nuclear magnetization will be a function of the position, and the spatial diffusion of the nuclear Zeeman energy is induced by flip-flop transitions due to DDIs between nuclear spins [1–3].

However, as is well known, the inner spin–spin interactions, both homonuclear and heteronuclear, can be changed by applying a rapidly oscillating radio-frequency (r.f.) irradiation [4]. Such techniques are extensively used to obtain high-resolution NMR spectra of solids by means of averaging the DDIs [5, 6]. For this purpose, various experimental techniques have been developed, many of which utilize pulsed r.f. fields. One of the first such experiments, based on pulse spin-locking [7], was carried out to study spin–lattice relaxation.

In the present paper, we study the spin diffusion and spin–lattice relaxation that are caused by coupling with PIs and under the influence of multiple-pulse r.f. irradiation.

2. The effective Hamiltonian

Let us consider a sample containing nuclear spins I coupled to PI spins S by DDIs and placed in a high constant magnetic field and multiple-pulse r.f. fields. The dynamics of the spin system in the frame, rotating with the nuclear Larmor frequency, can be described by a solution of the equation for the state operator $\rho(t)$:

$$\frac{d\rho(t)}{dt} = [\mathcal{H}(t), \rho(t)] \quad (1)$$

with the Hamiltonian

$$\mathcal{H}(t) = -f(t)I^x + \mathcal{H}_{II} + \mathcal{H}_{IS} + \mathcal{H}_S \quad (2)$$

where

$$f(t) = \omega_e \sum_{k=0}^{\infty} \delta\left(\frac{t}{t_c} - k - 1\right)$$

and where $\omega_e = \varphi/t_c$, t_c is the period of the multiple-pulse sequence, $\varphi = \gamma_I H_1 t_w$, γ_I is the gyromagnetic ratio of the nuclei, and H_1 is the amplitude of the r.f. pulse. \mathcal{H}_{II} is the secular part of the nuclear DDI Hamiltonian:

$$\mathcal{H}_{II} = \sum_{\mu \neq \eta} a_{\mu\eta} \left[I_{\mu}^z I_{\eta}^z - \frac{1}{4} (I_{\mu}^+ I_{\eta}^- + I_{\mu}^- I_{\eta}^+) \right] \quad (3)$$

where $a_{\mu\eta} = \gamma_I^2 r_{\mu\eta}^{-3} (1 - 3 \cos^2 \theta_{\mu\eta})$, and $r_{\mu\eta}$ and $\theta_{\mu\eta}$ are the spherical coordinates of the vector $\vec{r}_{\mu\eta}$ connecting the μ th and η th nuclei in a coordinate system with the Z -axis along the direction of the external magnetic field. In the impurity–nuclear DDI Hamiltonian, \mathcal{H}_{IS} , we retain also only the secular relative Z -axis term which gives the dominant contribution to the relaxation process:

$$\mathcal{H}_{IS} = \sum_{\mu j} b_{\mu j} I_{\mu}^z S_j^z \quad (4)$$

where $b_{\mu j} = \gamma_I \gamma_S r_{\mu j}^{-3} (1 - 3 \cos^2 \theta_{\mu j})$, γ_S is the gyromagnetic ratio of the PI, and $r_{\mu j}$ is the distance between nuclear and PI spins. Here, the Greek indices indicate the positions of the nuclei and the italic ones those of the impurities. \mathcal{H}_S describes the impurity spin system.

In order to take into account the effect of the multiple-pulse irradiation, we apply the unitary transformation

$$\tilde{\rho}(t) = U(t) \rho(t) U^+(t) \quad (5)$$

with the unitary operator

$$U(t) = \exp \left\{ -i \int_0^t dt' [f(t') - \omega_e] I^x \right\}. \quad (6)$$

The equation of motion of the state operator (1) in the transform frame is given by

$$i \frac{d\tilde{\rho}(t)}{dt} = [\tilde{\mathcal{H}}(t), \tilde{\rho}(t)] \quad (7)$$

where

$$\tilde{\mathcal{H}}(t) = \mathcal{H}_e + \mathcal{H}_{II}^0 + \sum_{m=-1, m \neq 0}^1 (\phi_{2m}(t) \mathcal{H}_{II}^{(2m)} + \phi_m(t) \mathcal{H}_{IS}^{(m)}) + \mathcal{H}_S \quad (8)$$

$$\mathcal{H}_e = \omega_e I^x. \quad (9)$$

Also, the functions

$$\phi_m(t) = \exp \left\{ -im \int_0^t dt' [f(t') - \omega_e] \right\}$$

are periodic with the period t_c . \mathcal{H}_{II}^0 is the secular part ($[\mathcal{H}_{II}^0, \mathcal{H}_e] = 0$) and $\mathcal{H}_{II}^{(2m)}$ is the non-secular part ($[\mathcal{H}_{II}^{(2m)}, \mathcal{H}_e] = 2m\omega_e \mathcal{H}_{II}^{(2m)}$) of the nuclear DDI Hamiltonian relative to the X -axis. $\mathcal{H}_{IS}^{(m)}$ is the non-secular part ($[\mathcal{H}_{IS}^{(m)}, \mathcal{H}_e] = m\omega_e \mathcal{H}_{IS}^{(m)}$) of the impurity–nuclear DDI Hamiltonian relative to the X -axis.

To take into account the influence of the time-dependent terms in equation (7), we expand the periodic functions $\phi_{2m}(t)$ and $\phi_m(t)$, with the period t_c , in Fourier series:

$$\phi_q(t) = \sum_{n=-\infty}^{\infty} c_n^{(q)} e^{-i\omega_n t} \quad \phi_q(t) = \phi_{-q}^*(t) \quad (10)$$

where

$$c_n^{(q)} = \frac{(-1)^n \sin \theta_q}{n\pi + \theta_q} \quad \theta_q = |q|\omega_e t_c / 2 \quad q = 2m, m \quad (11)$$

and write the Hamiltonian (7) in the following form:

$$\tilde{\mathcal{H}}(t) = \mathcal{H}_e + \mathcal{H}_{II}^0 + \sum_{n=-\infty}^{\infty} \sum_{m=-1, m \neq 0}^1 (c_n^{(2m)} \mathcal{H}_{II}^{(2m)} + c_n^{(m)} \mathcal{H}_{IS}^{(m)}) \exp(-im\omega_e t) + \mathcal{H}_S. \quad (12)$$

The value of t_c in multiple-pulse experiments is chosen such that $t_c \|\mathcal{H}_{II}\| \ll 1$ (here $\|\mathcal{H}_{II}\|$ is the norm of the nuclear DDI Hamiltonian), and to account for the oscillating nuclear DDI terms in equation (12) the Krylov–Bogolyubov–Mitropol'skii averaging method [8] can be used. The result of carrying out the averaging procedure on equation (12) may be written as an effective Hamiltonian:

$$\mathcal{H}_{eff}(t) = \mathcal{H}_e + \bar{\mathcal{H}}_{II} + \sum_{n=-\infty}^{\infty} \sum_{m=-1, m \neq 0}^1 c_n^{(m)} \mathcal{H}_{IS}^{(m)} \exp(-im\omega_e t) + \mathcal{H}_S \quad (13)$$

where

$$\bar{\mathcal{H}}_{II} = H_{II}^0 + A [\mathcal{H}_{II}^{-2}, \mathcal{H}_{II}^2] \quad (14)$$

$$A = \sum_{n=-\infty}^{\infty} \frac{c_n^{(2)} c_n^{(-2)}}{2\omega_n} = \frac{t_c}{2} \cot \theta_2. \quad (15)$$

3. The spin-diffusion equation

To obtain the equation describing the spin diffusion and spin–lattice relaxation of the spin system under the spin-locking excitation, we will use the method of non-equilibrium state operators [9], which has been applied to obtain the diffusion equation in the case of the Zeeman order spin diffusion in the laboratory frame [10]. Introducing a nuclear spin-density operator:

$$\vec{I}(\vec{r}) = \sum_{\mu} \delta(\vec{r} - \vec{r}_{\mu}) \vec{I}_{\mu} \quad (16)$$

the density of the operator \mathcal{H}_e can be written down in the form

$$\mathcal{H}_e(\vec{r}) = \omega_e I^x(\vec{r}). \quad (17)$$

Assuming that for a time $t > T_2$ (T_2 is a spin–spin relaxation time) after the beginning of the multiple-pulse irradiation a quasi-equilibrium state is established [11–13], then the state operator $\tilde{\rho}(t)$ can be written as

$$\rho = Z^{-1} \exp \left\{ - \int d\vec{r} \beta_e(\vec{r}, t) \mathcal{H}_e(\vec{r}) - \beta_S \mathcal{H}_S + \int_{-\infty}^0 dt e^{\varepsilon t} \left[\int d\vec{r} \beta_e(\vec{r}, t) \frac{\partial \mathcal{H}_e(\vec{r})}{\partial t} + \beta_S \frac{\partial \mathcal{H}_S}{\partial t} \right] \right\} \quad (18)$$

where $Z = \text{Tr} \exp\{\dots\}$ and the transition to the limit $\varepsilon \rightarrow +0$ should be made after the calculation of the integral. $\beta_e(\vec{r}, t)$ is the local inverse temperature of the nuclear dipole reservoir. In equation (18) all operators are taken in the Heisenberg representation. Taking into account that the heat capacity of the PI:

$$P_S = - \frac{\partial}{\partial \beta_S} \langle \mathcal{H}_S \rangle$$

is large in comparison with nuclear spin heat capacity ($P_S/P_I \sim \gamma_S^2/\gamma_I^2 \sim 10^6$) and that the spin–lattice relaxation time of the PI, T_{1S} , is very short ($T_{1S}/T_{1I} \sim 10^{-3}$) [14], a case which is experimentally realizable, it is reasonable to consider only the relaxation process with constant inverse spin temperature of the PI, β_S , equal to that of the lattice: $\beta_S = \beta_L$. Therefore, the PI's reservoir is in thermal equilibrium with the lattice and β_S is independent of the position.

Using the commutation rules for the components of the spin-density operator (17):

$$[I^x(\vec{r}), I^y(\vec{r}')] = i\delta(\vec{r} - \vec{r}')I^z(\vec{r}) \quad (19)$$

we can obtain the following equations in the form of localized laws of conservation of the spin energy densities:

$$\frac{\partial \mathcal{H}_e(\vec{r})}{\partial t} + \text{div } \vec{j}_e(\vec{r}) = K_{eS}(\vec{r}) \quad (20)$$

$$\frac{\partial \mathcal{H}_S}{\partial t} = - \int d\vec{r} K_{eS}(\vec{r}). \quad (21)$$

The last equation is the result of the energy conservation law. In equation (20), $\vec{j}_e(\vec{r})$ is the operator of the flux of the nuclear Zeeman energy in the effective field ω_e :

$$\begin{aligned} \vec{j}_e(\vec{r}) = \frac{i\omega_e}{2} \int d\vec{r}' (\vec{r} - \vec{r}') \left\{ \frac{1}{4} a(\vec{r} - \vec{r}') + 18A^2 \int d\vec{r}'' a(\vec{r} - \vec{r}'') a(\vec{r}'' - \vec{r}') \right. \\ \left. \times I^x(\vec{r}'') [I^+(\vec{r})I^-(\vec{r}') - I^-(\vec{r})I^+(\vec{r}')] \right\} \end{aligned} \quad (22)$$

and $K_{eS}(\vec{r})$ in equation (20) is the change of the nuclear Zeeman energy due to the interaction with the PI:

$$K_{eS}(\vec{r}) = \frac{i\omega_e}{2} \int d\vec{r}' b(\vec{r} - \vec{r}') [\phi_1(t)I^+(\vec{r}) - \phi_{-1}(t)I^-(\vec{r})] S^z(\vec{r}'). \quad (23)$$

An application of the state operator (18) to the averaging of the transport equation (20) in the high-temperature approximation results in the diffusion equation:

$$\frac{\partial \beta_e(\vec{r}, t)}{\partial t} = D \Delta \beta_e(\vec{r}, t) - T_1^{-1}(\vec{r}) [\beta_e(\vec{r}, t) - \beta_L] \quad (24)$$

with the boundary condition

$$\nabla \beta_e(r)|_{r=l} = 0 \quad (25)$$

where $l \sim (\gamma_S/\gamma_I)^{1/3} r_0$ is the radius of the diffusion barrier [1, 2], inside of which the spin diffusion process is quenched, and r_0 is distance between neighbouring nuclei. The first term on the right-hand side of equation (24) describes the variation (in time) of the dipolar order due to the spin diffusion with the diffusion coefficient

$$D = D^{(1)} + D^{(2)} \quad (26)$$

where $D^{(1)}$ is the spin-diffusion coefficient, computed to first order from the nuclear DDI Hamiltonian:

$$D^{(1)} = \frac{1}{4} \sum_{\mu \neq \eta} (\vec{r}_\mu - \vec{r}_\eta)^2 a^2(\vec{r}_\mu - \vec{r}_\eta) J_{\mu\eta}(\omega_e). \quad (27)$$

Here,

$$J_{\mu\eta}(\omega_e) = \int_{-\infty}^{\infty} dt g_{\mu\eta}(t) e^{i\omega_e t}$$

is the spectral function of the NMR resonance line with the correlation function

$$g_{\mu\eta}(t) = \langle I_{\mu}^{+}(0)I_{\eta}^{-}(t) \rangle. \quad (28)$$

$D^{(2)}$ is the second-order spin-diffusion coefficient:

$$D^{(2)} = (18A)^2 \sum_{\mu \neq \eta \neq \nu} (\vec{r}_{\mu} - \vec{r}_{\eta})^2 a^2 (\vec{r}_{\mu} - \vec{r}_{\nu}) a^2 (\vec{r}_{\eta} - \vec{r}_{\nu}) \int_{-\infty}^{\infty} dt g_{\mu\eta\nu}(t) e^{i\omega_e t} \quad (29)$$

with $g_{\mu\eta\nu}(t) = \langle (I_{\nu}^x)^2 I_{\mu}^{+}(0)I_{\eta}^{-}(t) \rangle$. Note that at $\omega_e t_c = \pi/2$ the second-order spin-diffusion coefficient $D^{(2)} = 0$.

The second term in equation (24) gives the rate of change of $\beta_e(\vec{r})$ due to the relaxation with the relaxation time $T_1(\vec{r})$ which is given, for a cubic crystal, by

$$T(\vec{r}) = \left(\frac{1}{2} \sum_{\mu j} b_{\mu j} J_j(\omega_e, \tau_c) \right)^{-1} \quad (30)$$

where τ_c is the correlation time and $J(\omega_e, \tau_c)$ is the spectral function:

$$J_j(\omega_e, \tau_c) = \frac{1}{2\pi} \sum_{n=-\infty}^{\infty} c_n^1 c_n^{-1} \int_0^{\infty} dt \operatorname{Re} \left[e^{-i(\omega_e + \omega_n)t} \langle S_j^z(0)S_j^z(t) \rangle \right]. \quad (31)$$

4. Results and discussion

Immediately after a disturbance of the nuclear spin system, there is no gradient of β_e , and diffusion cannot be of importance at the start of the relaxation process [15]. To describe the relaxation, we can use equation (24) without the first term; this is the so-called direct-relaxation regime [16]. In this case the normalized relaxation function:

$$R(t) = \frac{\beta(t) - \beta(\infty)}{\beta(0) - \beta(\infty)}$$

which is averaged over the sample takes the form [17]

$$R(t) = \exp \left[- \left(\frac{t}{T_1} \right)^{\alpha} \right]. \quad (32)$$

For a homogeneous distribution of paramagnetic centres and nuclear spins, $\alpha = Q/6$ where Q is the sample dimensionality; for an inhomogeneous distribution, the sample is divided into q -dimensional subsystems, each containing one paramagnetic centre, yielding $\alpha = (Q+q)/6$. If we assume that the local magnetization of the subsystem is constrained to taking a direction with respect to the effective field of $\vec{\omega}_e$, then the subsystem dimensionality $q = 1$. So, for a three-dimensional sample, $Q = 3$, the magnetization grows according to equation (32) with $\alpha = 2/3$, which is consistent with a spin-locking experiment [18] (figure 1). Furthermore, if we assume that in the high-temperature approximation the correlation function of the PI spins in equation (31) is exponential with correlation time τ_c :

$$\langle S_j^z(0)S_j^z(t) \rangle = \langle S_j^{z2}(0) \rangle \exp \left(- \frac{|t|}{\tau_c} \right) \quad (33)$$

then the dependence of T_1 on the correlation time τ_c and multiple-pulse spin-locking parameters t_c and φ can be found.

Substituting equation (33) in equation (31), we have

$$J_j(\omega_e, \tau_c) = \frac{1}{2\pi} \langle S_j^{z2}(0) \rangle \sum_{n=-\infty}^{\infty} c_n^1 c_n^{-1} \int_0^{\infty} dt \operatorname{Re} \left[e^{-i(\omega_n + \omega_e)t - |t|/\tau_c} \right]. \quad (34)$$

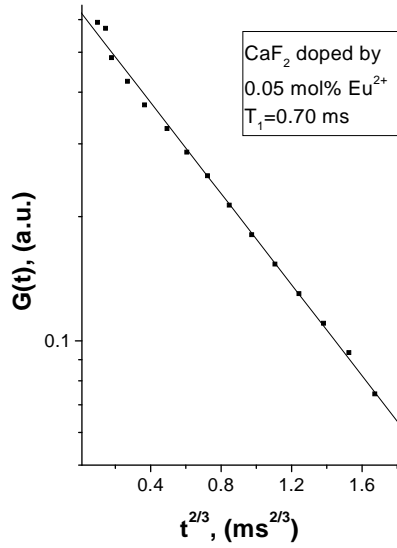


Figure 1. The log of the ^{19}F spin-locking signal in CaF_2 doped with Eu^{2+} (0.05 mol%) as a function of $t^{2/3}$ for the direct-relaxation regime. The solid line is an apparent fit to expression (32).

After integrating by parts twice and the summing over n , we obtain

$$J_j(\omega_e, \tau_c) = \frac{\tau_c}{2\pi} \left\langle S_j^z(0) \right\rangle \left[1 - \frac{\tau_c}{t_c} \frac{(1 - \cos \varphi) \sinh(t_c/\tau_c)}{\cosh(t_c/\tau_c) - \cos \varphi} \right]. \quad (35)$$

Using equations (30) and (35), the spin-lattice relaxation time for the direct-relaxation regime can be calculated as a function of the correlation time τ_c and multiple-pulse field parameters φ and t_c :

$$T_1 \sim \frac{1}{\tau_c} \left[1 - \frac{\tau_c}{t_c} \frac{(1 - \cos \varphi) \sinh(t_c/\tau_c)}{\cosh(t_c/\tau_c) - \cos \varphi} \right]^{-1}. \quad (36)$$

The direct-relaxation regime of the magnetization should be valid for a short time after a disturbance of the nuclear spin system [15]. Thus β_e is expected to start with a non-exponential time-dependent function, and proceed asymptotically to being an exponential function of time, in the so-called diffusion-relaxation regime [15, 16]. In this case we have to take into account also the first term in equation (24). One of the ways of solving this equation and extracting the time dependence is to introduce the eigenfunctions $\chi_n(\vec{r})$ of the operator $D\Delta - T^{-1}(\vec{r})$ [19]. Making the approximation of assuming spherical symmetry, the general solution of equation (24) can be written as an expansion in terms of the orthogonal functions $\chi_n(\vec{r})$:

$$\beta_d(r, t) = \int dr' \sum_n \exp(-k_n^2 D t) \chi_n(r) \chi_n^*(r') \quad (37)$$

where the functions $\chi_n(r)$ satisfy the equation

$$\Delta \chi_n(r) - \frac{T^{-1}(\vec{r})}{D} \chi_n(r) = -k_n^2 \chi_n(r) \quad (38)$$

with the boundary condition

$$\nabla \chi_n(r) \Big|_{r=l} = 0. \quad (39)$$

Equation (38) is well known in the theory of scattering for low-energy limit [20], and has an asymptotic solution for $r \gg l$, namely $\chi_n(r) \sim \sin(k_n r + \delta_n)/r$, where $\delta_n \sim k_n \xi$ is the phase shift and ξ is the scattering length [20]. In view of the spherical symmetry, only s-wave scattering with $n = 0$ is taken into account. Replacing the local spin–lattice relaxation time $T(\vec{r})$, equation (30), which is angularly dependent, by the average over angles $\theta_{\mu j}$, we obtain that $T^{-1}(\vec{r}) \sim B/r^6$, where

$$B \sim \gamma_I \gamma_S \langle J_j(\omega_e, \tau_c) \rangle_{\theta_{\mu j}}. \quad (40)$$

For $(B/D)^{1/4} \ll r \ll 1/k_0$, we obtain [20]

$$\xi = \left(\frac{B}{D} \right)^{1/4} \frac{\Gamma(3/4)}{2\Gamma(5/4)}. \quad (41)$$

Using the last result, for the long-time approximation $t \gg l^2/D$, we obtain the normalized relaxation function for the diffusion regime:

$$R(t) = \exp(-t/T_{1D}) \quad (42)$$

where

$$T_{1D} = \frac{1}{4\pi C_p D \xi^3}. \quad (43)$$

Substituting equation (41) in (43), we obtain an expression that describes the exponential time dependence of $\beta_e(t)$ with the relaxation time:

$$T_{1D} = \left[2\pi C_p D^{3/4} B^{1/4} \frac{\Gamma(3/4)}{\Gamma(5/4)} \right]^{-1} \quad (44)$$

which is inversely proportional to the impurity concentration C_p [14]. Using equations (35), (40), and (44), the spin–lattice relaxation time for the diffusion regime can be calculated as a function of the correlation time and multiple-pulse field parameters:

$$T_{1D} \sim \left(\frac{1}{\tau_c} \right)^{1/4} \left[1 - \frac{\tau_c}{t_c} \frac{(1 - \cos \varphi) \sinh(t_c/\tau_c)}{\cosh(t_c/\tau_c) - \cos \varphi} \right]^{-1/4}. \quad (45)$$

Fitting the experimental data [18] yields a good agreement with $T_{1D} = 1.75$ ms obtained for the diffusion part of the relaxation process (figure 2). At times longer than 2 ms, the direct-relaxation regime changes to the diffusion one.

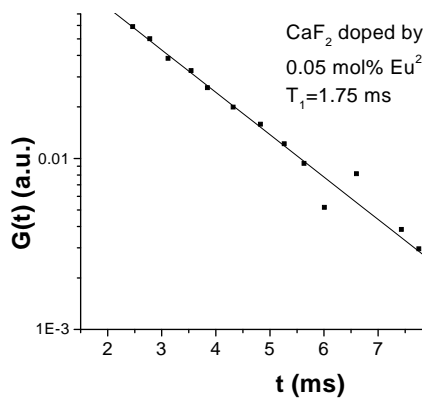


Figure 2. The log of the ^{19}F spin-locking signal in CaF_2 doped with Eu^{2+} (0.05 mol%) as a function of t for the diffusion-limited regime of relaxation. The solid line is an apparent fit to expression (42).

5. Conclusions

We obtained the spin-diffusion equation, which allows us to find the time dependence of the magnetization. The spin-lattice relaxation time is calculated as a function of the correlation time and multiple-pulse field parameters. At the beginning of the relaxation process the direct-relaxation regime is realized with non-exponential time dependence. This type of relaxation behaviour of the magnetization has been observed experimentally [18]. Then the relaxation regime will be changed to a diffusion one in accordance with $T_1 < T_{1D}$ [18].

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