Spin-Polarization Effects on the Phase Relaxation Induced by Dipole–Dipole Interactions

A. G. MARYASOV, S. A. DZUBA, AND K. M. SALIKHOV

Institute of Chemical Kinetics and Combustion, Novosibirsk—90, 630090, USSR

Spin-polarization effects on the phase relaxation due to dipole–dipole interactions of particles with 1/2 spin are studied theoretically for magnetically diluted solids. Paramagnetic centers with anisotropic g tensor are considered, random modulation of the dipole–dipole interaction by spin flip-flops in the process of spin–lattice relaxation (T_1 process) is taken into account. The T_1 process is shown to narrow the dipolar contour at the center of the line, the limiting dipolar width being proportional to the spin polarization p at $T_1 \rightarrow 0$. The angular dependence of the dipolar broadening is investigated. The signal decay kinetics are calculated for free induction and electron spin echo at arbitrary T_1 and p. Spin polarization is demonstrated to reduce the contribution from the dipole–dipole interaction to the irreversible spin dephasing by the spectral diffusion mechanism and to increase the contribution from the instantaneous diffusion mechanism to the echo signal decay.

INTRODUCTION

The present paper deals with the effect of spin polarization at low temperatures and the effect of g-tensor anisotropy upon the phase relaxation of unpaired electron spins of paramagnetic centers in magnetically diluted solids, induced by the dipole–dipole interaction between the PC spins. The contribution from the dipole–dipole interaction to the ESR spectral linewidth, to the free-induction (FI) signal decay and to the electron spin-echo (ESE) signal decay kinetics is considered.

There are many theoretical treatments of phase spin relaxation by dipole–dipole interactions. A static (inhomogeneous) broadening of an ESR line due to dipole–dipole interactions is discussed in Refs. (*1–3*). The dipole contour of ESR lines for centers with isotropic g tensors was shown to be described by the Lorentzian function with the half-width at the half-height for $S = 1/2$ equal to

$$\Delta\omega_{1/2} = (4\pi^2/g(3)^{1/2})9^2\beta^2\hbar^{-1}C, \qquad [1]$$

where β is the Bohr magneton, and C is the concentration of spins.

The temperature dependence of the static dipole broadening was studied by Altshuler and Mokeev (*3*). It was shown that $\Delta\omega_{1/2}$ is temperature independent for particles with a spin $S = 1/2$, and must increase with decreasing temperature for those with $S \geq 1$. Grinberg et al. (*4*) investigated the exchange narrowing of a static dipole contour.

In magnetically diluted solids ESR spectra are, as a rule, inhomogeneously broad-

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ened by the hyperfine interaction, the g-tensor anisotropy, etc. (5). This circumstance hampers measurements of the dipole broadening of the ESR spectra. The ESE method makes it possible to eliminate the disguising effect of the inhomogeneous broadening and to distinguish the contribution from a comparatively weak dipole–dipole interaction to the phase relaxation. The dipole–dipole interaction results in ESE signal decay by two mechanisms (6, 7): spectral diffusion and instantaneous diffusion. The theory of spectral and instantaneous diffusions was developed in a number of works reviewed in (8–11) with an exhaustive list of references.

The published theories of spin phase relaxation in magnetically diluted solids, induced by dipole–dipole interactions between centers refer to particles with an isotropic g tensor and to a high-temperature situation when the Zeeman energy of the unpaired electron is low compared to the thermal energy, and the spins are oriented along and against the external field with about the same probability. The only exception is paper (3), which considers spin-polarization effects on dipole broadening in a center with an isotropic g tensor. However, the authors discussed the static broadening only. Meanwhile, it is very interesting to study spin-polarization effects (at decreasing temperature) upon the dipole broadening of ESR lines and on the ESE signal decay under the condition that the local dipole field cannot be treated as a static one but varied randomly by spin flips. Note that the spin-polarization effect upon the ESE signal decay by the spectral diffusion mechanism in magnetically diluted systems has been already discussed, and some interesting preliminary estimates have been obtained (12–14).

An appreciable spin polarization arises at the following temperatures. Let $H_0 \approx 3300G$ be a typical field in ESR spectrometers. The temperature at which the Zeeman spin energy is comparable with the thermal energy is $T = g\beta H_0/k$. Hence, for particles with $g \cong 2$ we obtain $T = 0.4$ K. However, there are many paramagnetic ions with anisotropic g tensors (see, e.g. (12, 13)) whose effective g factor can reach several tens. For instance, at $g = 20$ spins are highly polarized even at liquid helium temperature.

The present paper is an attempt to give a systematic approach to spin-polarization effects on the phase relaxation induced by paramagnetic center dipole–dipole interactions in magnetically diluted solids. The centers are assumed to have anisotropic g tensors, the dipole–dipole interaction being modulated by random spin flips induced by spin–lattice interactions.

The basic assumptions of the theory are formulated in the second section. The third section deals with the effects of g tensor anisotropy and spin polarization on the dipole broadening and FI signal decay. The contribution from dipole–dipole interactions to the ESE signal decay by the mechanisms of spectral and instantaneous diffusion is discussed in the fourth section followed by a summary of the results obtained.

BASIC ASSUMPTIONS AND INITIAL EQUATIONS OF THEORY

Spin Hamiltonian of Dipole–Dipole Interactions

As noted above, in magnetically diluted solids the dipole broadening of ESR lines is much less than their inhomogeneous broadening. This fact enables one to take

into account only the adiabatic part of the dipole–dipole interaction Hamiltonian when considering spin phase relaxation processes (*1–11*). The nonadiabatic terms induce a shift of the center resonance frequencies only in the second order of perturbation theory and hence can be neglected for systems with a substantial broadening of the ESR spectra. Being responsible for the spin flips, these terms must be taken into account in discussing such processes as Zeeman energy transfer, spin diffusion, cross-relaxation (*8–10*).

Consider a system of paramagnetic centers with the spin Hamiltonian

$$\hat{\mathcal{H}} = \hat{\mathcal{H}}_z + \hat{\mathcal{H}}_{d-d} + \hat{\mathcal{H}}_{sl} .$$ [2]

Here the first term describes the Zeeman energy, the second corresponds to the spin d–d interaction, and the third is the spin–lattice interaction. For centers with anisotropic *g* tensors the Zeeman spin energy in an external field H_0 is (*15*)

$$\hat{\mathcal{H}}_z = \sum_i g_i \beta H_0 (\mathbf{k}_i \hat{\mathbf{S}}_i) = \sum_i g_i \beta H_0 \hat{S}_{ki} ,$$ [3]

where g_i is the effective *g* factor of the *i*th center, $\mathbf{k}_i$ is a unit vector in the direction of the effective magnetic field, and $\hat{S}_{ki}$ is the operator of projection of the *i*th spin moment onto the $\mathbf{k}_i$ direction which is a quantization axis. For a center with an axial symmetrical *g* tensor (*15*) we have

$$g_i = [g_{i\parallel}^2 \cos^2 \theta_i + g_{i\perp}^2 \sin^2 \theta_i]^{1/2} ,$$ [4]

where $g_\parallel$ and $g_\perp$ are the principal *g* values and θ_i is the angle between H_0 and the axis of symmetry. In the case of an axial symmetry without generality limitations, the following system of coordinates proves to be convenient. The axis of symmetry is the *Z* axis, the *X* axis being in the plane passing through the axis of symmetry and the external field direction. In this system the direction cosines of the effective field are (*15*)

$$\mathbf{k}_i \left(\frac{g_{i\perp}}{g_i} \sin \theta_i, 0, \frac{g_{i\parallel}}{g_i} \cos \theta_i \right).$$ [5]

To determine the adiabatic part, the total spin Hamiltonian must be written as the product of the spin projections onto the quantization axes:

$$\hat{\mathcal{H}}_{d-d}^a = \hbar \sum_{i>j} A_{ij} \hat{S}_{ki} \hat{S}_{kj} .$$ [6]

Let us write the expression for the dipole–dipole interaction parameter for the case though rather simple but encountered often in experiments with single crystals. Assume the *g* tensors of all the spins to be axially symmetric, the axes of symmetry being parallel. In this case,

$$A_{ij} = \frac{\beta^2 \hbar^{-1} r_{ij}^{-3}}{g_i g_j} \{ g_{i\perp}^2 g_{j\perp}^2 \sin^2 \theta + g_{i\parallel}^2 g_{j\parallel}^2 \cos^2 \theta$$

$$- 3[l \cdot g_{i\perp} g_{j\perp} \sin \theta + n \cdot g_{i\parallel} g_{j\parallel} \cos \theta]^2 \}, \quad [7]$$

where r_{ij} is the distance between the *i*th and *j*th particles, θ is the angle between H_0 and the *Z* axis, and *l* and *n* are the projections of the unit vector $\mathbf{r}_{ij}/r_{ij}$ to the *X* and

Z axis, respectively. Equation [7] can be written in the form

$$A_{ij} = \tilde{g}_{ij}^2 \beta^2 \hbar^{-1}(1 - 3 \cdot \cos^2 \psi_{ij})r_{ij}^{-3} . \qquad [8]$$

In Eq. [8], $\tilde{g}_{ij}^2$ is not merely the product of the effective g factors of interacting particles but

$$\tilde{g}_{ij}^2 \equiv [g_{i\perp}^2 g_{j\perp}^2 \sin^2 \theta + g_{i\parallel}^2 g_{j\parallel}^2 \cos^2 \theta]/g_i g_j . \qquad [9]$$

Hence, for similar centers we obtain

$$\tilde{g}^2 = (g_{\perp}^4 \sin^2 \theta + g_{\parallel}^4 \cos^2 \theta)/(g_{\perp}^2 \sin^2 \theta + g_{\parallel}^2 \cos^2 \theta). \qquad [10]$$

In Eq. [8], ψ_{ij} is the angle between $\mathbf{r}_{ij}$ and a certain direction in the plane passing through the external H_0 field direction and the axis of symmetry. This direction is set by the unit vector $\mathbf{q}$ with the projections

$$q_x = g_{i\perp} g_{j\perp} \sin \theta/(g_{i\perp}^2 g_{j\perp}^2 \sin^2 \theta + g_{i\parallel}^2 g_{j\parallel}^2 \cos^2 \theta)^{1/2} ,$$

$$q_y = 0, \qquad [11]$$

$$q_z = g_{i\parallel} g_{j\parallel} \cos \theta/(g_{i\perp}^2 g_{j\perp}^2 \sin^2 \theta + g_{i\parallel}^2 g_{j\parallel}^2 \cos^2 \theta)^{1/2} .$$

Note that in the case of an isotropic g tensor, $q_x = \sin \theta$, $q_z = \cos \theta$, i.e., $\mathbf{q}$ is directed along H_0. If only one tensor is isotropic, $\mathbf{q}$ is directed along the effective magnetic field of the partner.

In principle, the above method can be used to analyze the adiabatic part of dipole-dipole interactions in arbitrary situations: arbitrary g tensors, systems with various types paramagnetic centers, polycrystals, glasses, etc. However, we confine ourselves to single crystals containing either one or two types of centers with axially symmetric g tensors and parallel axes of symmetry.

Calculation Scheme for the Contribution of Dipole–Dipole Interactions to FI and ESE Signals

In the adiabatic approximation a dipole–dipole interaction simply shifts the spin resonance frequency. This fact makes it possible to write the contribution from the dipole–dipole interaction to the FI and ESE signal decays as *(1–11)*

$$v(t) = \left\langle \exp\left(-i \sum_j A(\mathbf{r}_j) \int_0^t s(t)m_j(t)dt\right)\right\rangle_{r,t} , \qquad [12]$$

where $s(t) = 1$ for FI, $s(t) = -1$ in the time interval $(0, \tau)$ and $s(t) = 1$ in the interval $(\tau, 2\tau)$ for ESE; in the latter case $t = 2\tau$, τ is the time interval between two microwave pulses. In Eq. [12], m_j is the spin projection of the jth partner on the quantization axis, $\langle \cdot \cdot \cdot \rangle_{r,t}$ means averaging over all random distributions of spin partners in the lattice ($\langle \cdot \cdot \cdot \rangle_r$) and over all random realizations of the process $m_j(t)$ ($\langle \cdot \cdot \cdot \rangle_t$). In averaging over all possible spin orientations it is necessary to take into account also the spin flips induced by microwave pulses forming the FI and ESE signals.

In magnetically dilute systems it can be assumed with good approximation that different spins flip due to spin–lattice interactions independently. Hence, Eq. [12]

can be reduced to

$$v(t) = \left\langle \prod_j \left\langle \exp(-iA(\mathbf{r}_j) \int_0^t s(t)m_j(t)dt) \right\rangle_t \right\rangle_r = \left\langle \prod_j v(t|\mathbf{r}_j) \right\rangle_r, \qquad [13]$$

where $v(t|\mathbf{r})$ is the contribution of a partner at distance r:

$$v(t|\mathbf{r}) = \left\langle \exp(-iA(\mathbf{r}) \int_0^t s(t)m(t)dt) \right\rangle_t. \qquad [14]$$

In the case of a homogeneous distribution of centers, the averaging of Eq. [13] by the spatial location results in (1–11)

$$v(t) = \exp[-C \int (1 - v(t|\mathbf{r}))d^3\mathbf{r}], \qquad [15]$$

where C is the concentration of paramagnetic centers. Then $v(t)$ can be calculated in two different ways. First, one can solve the kinetic equation for the density matrix of a pair of spins, find $v(t|\mathbf{r})$, and substitute the result obtained into Eq. [15]. The merit of this calculation scheme lies in the fact that in extreme cases it is possible to obtain exact asymptotic relations. However, in the general case, the integral in Eq. [15] can be calculated only numerically. Second, the averaging over the spatial distribution of centers can be performed prior to that over the random process $m(t)$. This calculation scheme has been proposed (16) as applied to a high-temperature limit.

From Eqs. [14] and [15] we obtain

$$v(t) = \exp\{-C \left\langle \int d^3\mathbf{r}(1 - \exp(-iA(\mathbf{r})f(t))) \right\rangle_t\}, \qquad [16]$$

with the random quantity

$$f(t) = \int_0^t s(t)m(t)dt. \qquad [17]$$

Substituting the expression for $A(\mathbf{r})$ and integrating over $d^3\mathbf{r}$, we obtain

$$v(t) = \exp\left\{-\frac{8\pi^2}{9(3)^{1/2}} \tilde{g}^2\beta^2\hbar^{-1}C\langle|f(t)|\rangle_t - iC \int d^3\mathbf{r}\langle\sin(A(\mathbf{r})f(t))\rangle_t\right\}. \qquad [18]$$

The real part of the exponent in Eq. [18] describes the FI and ESE signal decays due to dipole–dipole interactions, the imaginary part corresponds to changes in the spin precession phase in a mean local (dipole) field. It will be shown below that the imaginary part of $v(t|r)$ is indeed proportional to the mean polarization of the sample,

$$p = (1/2)g\beta C \cdot \tanh(g\beta H_0/2kT). \qquad [19]$$

As a result of the spin precession in the mean local field, the ESE signal acquires a phase since the macroscopic polarization of the sample (and thus the mean local field) is different within the time intervals $(0, \tau)$ and $(\tau, 2\tau)$. A detailed analysis of the precessing magnetization phase at the moment of the ESE signal formation could be informative of the mean local field. The mean local field is by no means manifested if the first microwave pulse, inducing the echo, turns the sample mag-

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netization through 90°. Indeed, in this case the macroscopic magnetization of the sample goes to zero within the time T_2^* (FI signal decay time).

If the mean local field is inhomogeneous throughout the species, the echo signals from a different part of the species have different phases, which results in an additional decay of the total ESE signal from the whole sample. Analysis of this decay might convey information on inhomogeneities of the mean fields. We will not discuss in more detail the very interesting problem of mean local fields (see, e.g. (17, 18)).

Thus, the FI and ESE decays due to dipole–dipole interactions obey the equations

$$v(t) = \exp[-C \int (1 - \text{Re}\{v(t|\mathbf{r})\})d^3\mathbf{r}],$$ [20a]

$$v(t) = \exp\left[-\frac{8\pi^2}{9(3)^{1/2}} \tilde{g}^2\beta^2\hbar^{-1}C\langle|f(t)|\rangle_t\right].$$ [20b]

The mean $|f(t)|$ value in Eq. [20b] was calculated numerically by simulating the random spin flip dynamics by the Monte Carlo technique. The quantity $v(t|\mathbf{r})$ was obtained in an analytic form. All computations were performed for a center with $S = 1/2$. In the Monte Carlo calculations we used a standard approach to the simulation of the Poisson process (19).

Calculations of $v(t|\mathbf{r})$ for a Particle with $S = 1/2$

This quantity can be readily found in the static limit when random spin flips can be neglected. The equilibrium density matrix of a pair of spins is

$$\rho_0 = Z^{-1}\exp[-(g_1\beta H_0\hat{S}_{1k_1} + g_2\beta H_0\hat{S}_{2k_2})/kT],$$

$$Z = \text{Tr}\{\exp[-(g_1\beta H_0\hat{S}_{1k_1} + g_2\beta H_0\hat{S}_{2k_2})/kT]\}.$$ [21]

The spin rotations round the Y axis by the microwave pulses inducing FI and ESE signals are set by the operator

$$\hat{\tilde{Q}} = \exp[-i(\theta_1\hat{S}_{1y} + \theta_2\hat{S}_{2y})].$$ [22]

Immediately after the first pulse the density matrix is

$$\rho(0) = \hat{\tilde{Q}}\rho_0\hat{\tilde{Q}}^{-1} \equiv \hat{Q}\rho_0,$$ [23]

where $\hat{Q}$ is the flip operator in Liouville's representation. If the pulse turns the spin through θ, subsequent to the pulse the state populations conforming to different orientations are

$$\rho_{1/2,1/2}(0) = \left[q\cos^2\frac{\theta}{2} + \sin^2\frac{\theta}{2}\right]\bigg/(1+q),$$

$$\rho_{-1/2,-1/2}(0) = \left[\cos^2\frac{\theta}{2} + q\sin^2\frac{\theta}{2}\right]\bigg/(1+q),$$ [24]

where

$$q = \exp(-g\beta H_0/kT).$$ [25]

Averaging Eq. [14] over the initial states [24], we obtain the following result for the FI signal

$$v_{FI}(t|\mathbf{r}) = \cos(At/2) + i \tanh(g\beta H_0/2kT) \cos\theta \sin(At/2). \qquad [26]$$

Hence, in the high-temperature limit

$$v_{FI}(t|\mathbf{r}) = \cos(At/2), \qquad [27]$$

which describes the FI signal modulation by the dipole–dipole spin interaction in a pair. Note that the imaginary part $v(t)$ in [26] is proportional to the equilibrium spin polarization,

$$\tanh(g\beta H_0/2kT) = (1 - q)/(1 + q). \qquad [28]$$

Likewise, in the static limit, from Eq. [14] we find that the dipole–dipole interaction of a spin with its partner contributes to the ESE signal of this spin as follows:

$$v_{ESE}(2\tau|\mathbf{r}) = \cos^2\frac{\theta^{(2)}}{2} + \sin^2\left\{\frac{\theta^{(2)}}{2}\right\}\cos(A\tau)$$

$$-i \tanh(g\beta H_0/2kT) \cos\theta^{(1)} \sin^2\left\{\frac{\theta^{(2)}}{2}\right\}\sin(A\tau), \qquad [29]$$

where $\theta^{(1)}$ and $\theta^{(2)}$ are the spin-partner rotation angles after the first and the second pulses. The imaginary part $v(t|\mathbf{r})$ is again seen to be proportional to the mean polarization. In the high-temperature limit we obtain the known result (11):

$$v_{ESE}(2\tau|\mathbf{r}) = \cos^2\frac{\theta^{(2)}}{2} + \sin^2\frac{\theta^{(2)}}{2}\cos A\tau. \qquad [30]$$

In the general case, when the random spin flips average the dipole–dipole interaction, $v(t|\mathbf{r})$ is calculated by kinetic equations for the density matrix of the spin pair.

Changes in the density matrix of a pair of centers induced by the dipole–dipole interaction and by random flips obey the equation

$$\frac{\partial\rho}{\partial t} = -i[A\hat{S}_{1k_1}\hat{S}_{2k_2}, \rho] + \hat{R}\rho. \qquad [31]$$

The form of the relaxation operator $\hat{R}$ in the high-temperature limit already has been discussed (11). At an arbitrary temperature the form of $\hat{R}$ can be found as proposed in Ref. (20). A formal solution of Eq. [31] can be given as

$$\rho(t) = \hat{L}(t)\rho(0). \qquad [32]$$

For S = 1/2, write the kinetic equations for the matrix ρ elements in the basis of states

$$|1\rangle = |\alpha_1\alpha_2\rangle, \qquad |2\rangle = |\alpha_1\beta_2\rangle, \qquad |3\rangle = |\beta_1\alpha_2\rangle, \qquad |4\rangle = |\beta_1\beta_2\rangle,$$

where α_i and β_i are the eigenstates of the operator $\hat{S}_{ik_i}$ conforming to the eigenvalues $+1/2$ and $-1/2$. According to (11, 20), we obtain the following equations, e.g., for

ρ_{13} and ρ_{24};

$$\frac{\partial \rho_{13}}{\partial t} = -i\frac{A}{2}\rho_{13} - W_2\rho_{13} - \frac{1}{2}W_1(1 + q_1)\rho_{13} + W_2q_2\rho_{24},$$

$$\frac{\partial \rho_{24}}{\partial t} = i\frac{A}{2}\rho_{24} - W_2q_2\rho_{24} - \frac{1}{2}W_1(1 + q_1)\rho_{24} + W_2\rho_{13}, \qquad [33]$$

where W_i is the mean rate of a random flip of the ith spin from $|\alpha\rangle$ to $|\beta\rangle$ state, i.e., W_i^{-1} is the mean lifetime of the ith spin in the excited state; $W_i^{-1}q_i^{-1}$ is the mean lifetime of the ith spin in the ground, unexcited state, with

$$q_i = \exp(-g_i\beta H_0/kT). \qquad [34]$$

The matrix elements ρ_{13} and ρ_{24} describe variations of the transverse magnetization of spin 1. In Eq. [33] the terms

$$-\frac{1}{2}W_1(1 + q_1)\rho_{13}, \qquad -\frac{1}{2}W_1(1 + q_1)\rho_{24}$$

describe the transverse relaxation of spin 1 due to its own random flips. The terms of Eq. [33] proportional to W_2 characterize random changes of the dipole frequency shift of spin 1 resulting from spin-2 flips. Quite analogous equations can be written for ρ_{12} and ρ_{34} determining the transverse magnetization of spin 2.

The characteristic numbers of Eq. [33] are

$$\lambda_{1,2} = -\frac{W_1(1 + q_1)}{2} - \frac{W_2(1 + q_2)}{2} \pm (R_{2+} + iR_{2-}),$$

where

$$R_{2+} = (1/2(2)^{1/2})\ \mathrm{sign}\ (A)\{[(W_2^2(1 + q_2)^2 - A^2)^2 + 4W_2^2A^2(1 - q_2)^2]^{1/2}$$

$$+\ W_2^2(1 + q_2)^2 - A^2\}^{1/2},$$

$$R_{2-} = (1/2(2)^{1/2})\{[(W_2^2(1 + q_2)^2 - A^2)^2 + 4W_2^2A^2(1 - q_2)^2]^{1/2}$$

$$-\ W_2^2(1 + q_2)^2 + A^2\}^{1/2}. \qquad [35]$$

The solution of Eq. [33] is expressed by the relations

$$\begin{pmatrix} \rho_{13}(t) + \rho_{24}(t) \\ \rho_{13}(t) - \rho_{24}(t) \end{pmatrix} = \exp\left(-\frac{W_1(1 + q_1)t}{2}\right)\begin{pmatrix} L_{01}L_{02} \\ L_{03}L_{04} \end{pmatrix}\begin{pmatrix} \rho_{13}(0) + \rho_{24}(0) \\ \rho_{13}(0) - \rho_{24}(0) \end{pmatrix}, \qquad [36]$$

where

$$L_{01} = \kappa[\cosh(R_2 t) + (W_2(1 + q_2)/2R_2)\sinh(R_2 t)],$$

$$L_{02} = -i\kappa(A/2R_2)\sinh(R_2 t),$$

$$L_{03} = i\kappa[(2R_2 - W_2^2(1 + q_2)^2/2R_2)/A]\sinh(R_2 t), \qquad [37]$$

$$L_{04} = \kappa[\cosh(R_2 t) - (W_2(1 + q_2)/2R_2)\sinh(R_2 t)],$$

$$R_2 = R_{2+} + iR_{2-}, \qquad \kappa = \exp(-W_2(1 + q_2)t/2).$$

In evolution operator [36] the term $\exp(-W_1(1 + q_1)t/2)$ describes the proper phase relaxation of spin 1. The operator $\hat{L}_0$ corresponds to the contribution from the partner to the relaxation of spin 1.

Using the operators $\hat{Q}$ and $\hat{L}_0$ that describe the spin motion induced by the microwave pulses (which form the signals observed) and the contribution from the dipole–dipole interaction to the spin dynamics, we obtain for the FI and ESE:

$$\rho_{FI}(t) = \hat{L}_0(t)\hat{Q}_1\rho_0 ,$$

$$\rho_{ESE} = \hat{L}_0(\tau)\hat{Q}_2\hat{L}_0(\tau)\hat{Q}_1\rho_0 , \qquad [38]$$

where $\hat{Q}_1$ and $\hat{Q}_2$ are the operators of spin-2 flips by the microwave pulses at time moments $t = 0$ and $t = \tau$, respectively.

As a result, we have the following equations for the FI and ESE signal decays. For the FI of a chosen spin:

$$\mathrm{Re}\{v_{FI}(t|\mathbf{r})\} = \exp(-W(1 + q)t/2)\{\cosh(R_+t) \cos (R_-t)$$

$$+ (1/2(R_+^2 + R_-^2))[(W(1 + q)R_+ + AR_- \tanh (g\beta H_0/2kT)$$

$$\times \cos \theta^{(1)}) \sinh(R_+t) \cos (R_-t) + (W(1 + q)R_- - AR_+ \tanh$$

$$\times (g\beta H_0/2kT) \cos \theta^{(1)}) \cosh (R_+t) \sin (R_-t)]\}, \qquad [39]$$

where W, q, $R\pm$, g, θ are referred to a partner. For the ESE:

$$v_{ESE}(2\tau|\mathbf{r}) = (1/2) \exp\{-W(1 + q)\tau\}\{\cosh (R_+\tau) + \cos (2R_-\tau)$$

$$+ [(W^2(1 + q)^2 + A^2 \cos \theta^{(2)})/4(R_+^2 + R_-^2)][\cosh (2R_+\tau)$$

$$- \cos (2R_-\tau)] + [W(1 + q)/(R_+^2 + R_-^2)][R_+ \sinh (2R_+\tau)$$

$$+ R_- \sin (2R_-\tau)] + A \tanh (g\beta H_0/2kT) \cos \theta^{(1)} \cos^2 \frac{\theta^{(2)}}{2}$$

$$\times (R_- \sinh (2R_+\tau) - R_+ \sin (2R_-\tau))/(R_+^2 + R_-^2)\}. \qquad [40]$$

Substitute Eq. [39, 40] into Eq. [20a] and obtain the FI and ESE signal decays due to the dipole–dipole interaction between centers. The Fourier transformation of the FI signal gives the dipole contour of the ESR line and the dipole line broadening.

FI SIGNAL DECAY AND ESR LINE BROADENING

Static Limit

In the static limit Eqs. [20] and [26] yield the following expression for the FI signal decay kinetics

$$v_{FI}(t) = \exp\{-\Delta\omega_{1/2}|t|\}, \qquad [41]$$

where $\Delta\omega_{1/2}$ is dipole broadening (see, e.g. [1]), and the dipole contour of the ESR line is described by the Lorentzian

$$g(\omega) = (1/\pi)\cdot \Delta\omega_{1/2}/(\omega^2 + \Delta\omega_{1/2}^2), \qquad [42]$$
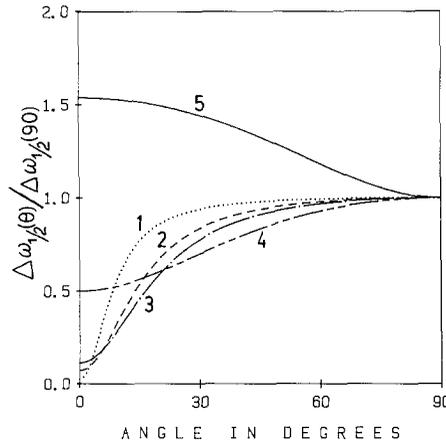
FIG. 1. Angular dependence of dipole broadening in single crystals containing paramagnetic centers of the same type. (1) Er^{3+} in $CaWO_4$, $K = 45$; (2) Yb^{3+} in $CaWO_4$, $K = 14$; (3) Fe^{3+} in $PbTiO_3$, $K = 8.83$; (4) Co^{2+} in $CaCO_3$, $K = 2.0$; (5) Ni^+ in LiF, $K = 0.65$.

where ω is the frequency deviation from resonance. The results [41] and [42] are well known for a center with an isotropic g tensor $(1-3)$. For particles with anisotropic g tensor the dipole broadening is described by the following relation.

The dipole broadening due to interactions of centers of the same type is (see Eq. [10])

$$\Delta\omega_{1/2} = \frac{4\pi^2}{9(3)^{1/2}} \tilde{g}^2\beta^2\hbar^{-1}C, \tag{43}$$

$$\tilde{g}^2 = (g_\perp^4 \sin^2\theta + g_\parallel^4 \cos^2\theta)/(g_\perp^2 \sin^2\theta + g_\parallel^2 \cos^2\theta). \tag{44}$$

In Eq. [44], θ is the angle between the axis of symmetry and the field direction H_0.

The dipole line broadening of paramagnetic centers of one type (marked by 1) due to the dipole–dipole interactions with centers of some other type (marked by 2) in the case, when the axes of symmetry of both species are parallel, is (see Eq. [9])

$$\Delta\omega_{1/2} = \frac{4\pi^2}{9(3)^{1/2}} \tilde{g}_{12}^2\beta^2\hbar^{-1}C, \tag{45}$$

where

$$\tilde{g}_{12}^2 = (g_{1\perp}^2 g_{2\perp}^2 \sin^2\theta + g_{1\parallel}^2 g_{2\parallel}^2 \cos^2\theta)/g_1 g_2 , \tag{46}$$

and g_1 and g_2 are effective g factors [4].

Figure 1 shows the angular dependence of $\Delta\omega_{1/2}$ for several $K = (g_\perp/g_\parallel)^2$ conforming to different particular systems (21). Note that the angular dependence of the dipole broadening is not merely determined by the product of the effective g values of the two centers [4]. The real angular dependence of $\Delta\omega_{1/2}$ is *stronger* than that of the product of the effective g values. Figure 2 illustrates this statement by curves showing a deviation of $\tilde{g}^2$ from g_{eff}^2. These results agree with experimental data available for the angular dependence of dipole broadening for Er^{3+} ions in
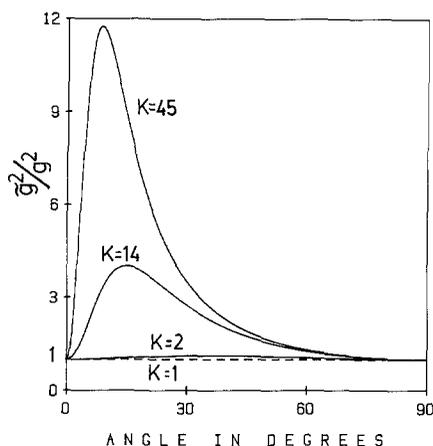
FIG. 2. $\tilde{g}^2$ Deviation from a simple product of effective g values of two paramagnetic centers.

$CaWO_4$ (22). Note that in the static limit the ESR dipole linewidth is independent of spin polarizations for $S = 1/2$. This result has been obtained earlier (3) and differs fundamentally from that known for magnetoconcentrated solids (23). The second moment of the dipolar frequency shift decreases with falling temperature (23),

$$\langle \Delta\omega_{d-d}^2 \rangle \sim 1 - \tanh^2 (g\beta H_0/2kT). \qquad [47]$$

The fact that in magnetodilute solids the static dipole broadening does not go to zero with falling temperature can be interpreted as follows. As known, in dilute systems the spin environment of different paramagnetic centers differs essentially. Therefore, even at a full polarization of the spins, local dipole fields differ strongly at different centers, i.e., a spread of local fields (dipole ESR line broadening) is preserved even at a complete polarization of spins. However, the spin polarization manifests itself in a shift of the line centre.

Averaging of Dipole–Dipole Interactions by Random Spin Reorientations

Random spin flips modulate dipole–dipole interactions, averaging them (6–11). Let us analyze the way the random spin flips affect the FI signal and dipole ESR line contour.

First of all consider a pair of spins. The frequency of the spin considered is either $-A/2$ or $+A/2$ shifted by the partner, depending on the orientation of the latter. Random spin flips result in exchange between these two frequency shifts. At a sufficiently high reorientation rate, when $W > |A|$, the spectrum becomes exchange-narrowed, two lines at the frequencies $\pm A/2$ are replaced by one with an average frequency shift equal to

$$\overline{\Delta\omega(\mathbf{r})} = -(A(\mathbf{r})/2) \tanh (g\beta H_0/2kT). \qquad [48]$$

In the general case, the pair problem is solved by Eq. [39]. At a fast exchange,
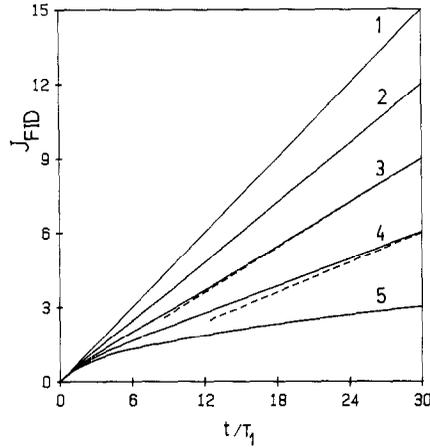
FIG. 3. Time evolution of J_{FID} for different spin-polarization degrees: (1) $p = 1$, (2) $p = 0.8$, (3) $p = 0.6$, (4) $p = 0.4$, (5) $p = 0$. At sufficiently long times the curves conforming to $p \neq 0$ are asymptotically described by Eq. [53] (shown by dashed lines for $p = 0.4$ and 0.6).

$W \gg |A|$, Eq. [39] yields for the time range $t > T_1$

$$\text{Re}\{v_{FI}(t|\mathbf{r})\} \approx \exp[-M_2(\mathbf{r})T_1 t]\cos(\overline{\Delta\omega(\mathbf{r})} \cdot t). \qquad [49]$$

Here T_1 is the spin–lattice relaxation time equal to (20)

$$T_1 = 1/W(1 + q); \qquad [50]$$

M_2 is the spin frequency shift dispersion due to the dipole–dipole interaction with a partner,

$$M_2 = A^2(1 - \tanh^2(g\beta H_0/2kT))/4. \qquad [51]$$

Note that Eq. [51] includes the same temperature factor as Eq. [47].

In the limit of a very high W, Eq. [49] yields

$$\text{Re}\{v_{FI}(t|\mathbf{r})\} \approx \cos(\overline{\Delta\omega(\mathbf{r})} \cdot t). \qquad [52]$$

This result can be definitely interpreted. Under exchange narrowing the spin considered precesses in the average field of the partner. If the condition of fast exchange is met for all partners in the species, by substituting Eq. [52] into Eq. [20a] we obtain the following asymptotic relation for the FI decay kinetics ($W \to \infty$)

$$v_{FI}(t) = \exp\{-\Delta\omega_{1/2}|t| \tanh(g\beta H_0/2kT)\}. \qquad [53]$$

At a high temperature, when the spin polarizations are negligible, the FI exponential decay [53] does not manifest itself. In this case ($T \to \infty$, $q \to 1$), Eq. [49] gives

$$\text{Re}\{v_{FI}(t|\mathbf{r})\} \approx \exp\{-A^2 t/8W\}. \qquad [54]$$

The contribution from the dipole–dipole interaction with all centers, according to

Eqs. [54] and [20a] obeys the equation obtained also in Ref. (4)

$$v_{\mathrm{FI}}(t) \approx \exp(-a(t)^{1/2}),$$ [55]

$$a = \frac{8\pi(\pi)^{1/2}}{9(3)^{1/2}} \tilde{g}^2\beta^2\hbar^{-1}C(T_1)^{1/2}, \qquad T_1 \equiv 1/2W.$$ [56]

In the general case of arbitrary temperature and random spin flips, to analyze the FI decay kinetics one has to make numerical calculations by either Eqs. [20a] and [39] or Eq. [20a]. The FI decay kinetics can be expressed via a universal function J_{FID} which depends on two dimensionless parameters: $tW(1 + q) = t/T_1$ and the degree of spin polarization,

$$p = \tanh\,(g\beta H_0/2kT).$$

According to Eq. [20], the FI signal can be written as

$$v_{\mathrm{FI}}(t) = \exp\{-2\Delta\omega_{1/2}T_1 J_{\mathrm{FID}}(p, t/T_1)\}.$$ [57]

Figure 3 shows numerical calculations of J_{FID}. Curve 1 coincides with Eq. [41] and conforms to the case of a complete spin polarization; curve 5 corresponds to the high-temperature limit [55]. For transient temperatures the curves of Fig. 3 follow Eq. [53] at $t \gg T_1$. The tangent of the slope of the straight line at $t \gg T_1$ varies proportionally to the polarization degree p.

At $t < T_1$

$$J_{\mathrm{FID}} \approx t/2T_1 - (1/8)(1 - p^2)(t/T_1)^2.$$

Figures 4 and 5 illustrate the way random spin flips affect the dipole line contour. The dipole contour is seen to be narrowed by random spin flips. In the high-temperature limit (Fig. 4, see also (4)) the dipole contour shape cannot be described by
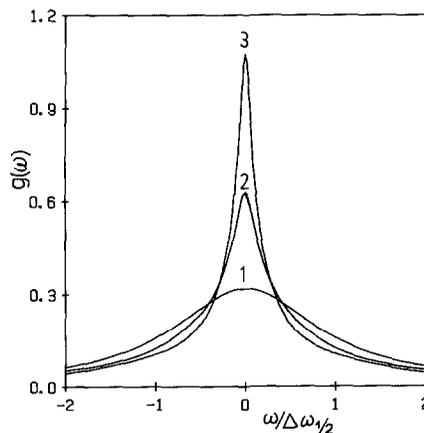
FIG. 4. Plots of the dipole contour of ESR lines, expressed through dimensionless variables in the high-temperature limit, vs the spin–lattice relaxation time. Different curves conform to different values of the parameter $R = \Delta\omega_{1/2}T_1$. (1) $R \to \infty$ static limit, the Lorentz distribution, (2) $R = 1$, (3) $R = 0.5$. As the spin relaxation flip rate increases, the line narrows at $R \approx 1$.
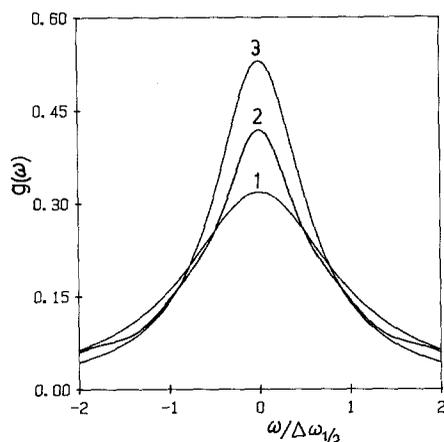
FIG. 5. Plots of the dipole contour vs the spin–lattice relaxation time ($R = \Delta\omega_{1/2}T_1$) at $p = 0.6$: (1) $R \to \infty$, (2) $R = 1$, (3) $R = 0$.

the Lorentz function [42]: at the centre the line is narrower than the Lorentz function, and at the wings it falls as the Lorentz distribution with $\Delta\omega_{1/2}$ half-width. As the relaxation flip rate increases, the line half-width at half-height tends to zero. The situation differs for falling temperature. As the random flip rate grows, the line half-width goes to (see Eq. [53])

$$\Delta\Omega_{1/2} = \Delta\omega_{1/2} \tanh (g\beta H_0/2kT) = p\Delta\omega_{1/2} . \qquad [58]$$

At the center the narrowed line obeys the Lorentz distribution with $\Delta\Omega_{1/2}$ half-width, at the wings it decreases gradually in line with the Lorentz distribution with $\Delta\omega_{1/2}$ half-width (see Fig. 5). Remember that the line contour is obtained by the Fourier transformation of the FI decay.

CONTRIBUTION FROM DIPOLE–DIPOLE INTERACTION TO THE ESE SIGNAL DECAY

Static Limit

It is well known that in the static limit ($W \to 0$) dipole–dipole interactions contribute to the ESE signal decay only by the mechanism of so-called instantaneous diffusion (6–11).

Equations [29] and [20a] yield

$$v_{\text{ESE}}(2\tau) = \exp\left[-\left\langle \sin^2 \frac{\theta^{(2)}}{2} \right\rangle_g \Delta\omega_{1/2} \cdot 2\tau \right], \qquad [59]$$

which coincides with the known (9–11) expression for the ESE signal decay by the instantaneous diffusion mechanism at high temperatures. Since the dipole broadening for centers with $S = 1/2$ is temperature independent, the contribution from the instantaneous diffusion mechanism does not depend on the spin polarization either. In Eq. [59], $\langle \cdot \cdot \cdot \rangle_g$ means averaging over the rotation angles of spins with different resonance frequencies (9–11).

The Case of Very High W

In the other extreme case of very high rates of random spin flips, dipole–dipole interactions between centers contribute to the ESE signal decay only by the mechanism of spectral diffusion (6–9). At a high W ($W > |A|$, $W\tau > 1$) we obtain from Eq. [40]

$$v_{\rm ESE}(2\tau|{\bf r}) \approx \exp\{-A^2({\bf r})(1 - p^2)\tau/2W(1 + q)\}.$$ [60]

Averaging over all possible paramagnetic center spatial distributions according to Eq. [20a] gives

$$v_{\rm ESE}(2\tau) \approx \exp(-a_1(\tau)^{1/2}),$$ [61]

where

$$a_1 = \frac{8\pi(2\pi)^{1/2}}{9(3)^{1/2}} \tilde{g}^2\beta^2\hbar^{-1}C[T_1(1 - p^2)]^{1/2}, \qquad T_1 \equiv 1/W(1 + q).$$ [62]

Hence, the spin polarizations are seen to decrease the contribution from spectral diffusion to the spin dephasing, $a_1 \to 0$ at $p \to 1$. At a high W the d–d interaction contribution to the ESE signal decay is determined by the dispersion of local fields (see Eqs. [51] and [60]), a fact which results in the term $1 - p^2$ in Eqs. [60]–[62] (cf. Eq. [47]).

Arbitrary Temperatures and Arbitrary W

In the general case, the ESE signal decay due to d–d interactions follows Eqs. [20] and [40]. The phase relaxation of a definite spin depends on the angles of spin-partners rotations induced by the microwave pulses forming ESE signals (see Eq. [41]). To begin with, consider a system of two species of spins, A and B. Let us assume that microwave pulses affect only spins A, and find the contribution of spins B to the spin A echo signal decay.

This situation often occurs in ESE experiments (6–11). Moreover, just this case is of great interest in investigations of spin-polarization effects on phase relaxation (12, 13). The contribution of a spin B to the echo signal of a spin A is described by Eq. [40], the rotation angles of the spin B assumed to be zero. As a result,

$$v_{\rm B}(2\tau|{\bf r}) = (1/2) \exp(-\tau/T_1)\{\cosh(2R_+\tau) + \cos(2R_-\tau) + [(T_1^{-2} + A^2)/4(R_+^2 + R_-^2)]$$

$$\times (\cosh(2R_+\tau) - \cos(2R_-\tau)) + [T_1^{-1}/(R_+^2 + R_-^2)](R_+ \cdot \sinh(2R_+\tau)$$

$$+ R_- \sin(2R_-\tau)) + Ap(R_- \sinh(2R_+\tau) - R_+ \sin(2R_-\tau))/(R_+^2 + R_-^2)\}.$$ [63]

In the case of a uniform distributions of B spins in the sample, their total contribution to the A ESE signal decay is described by the expression obtained by substituting Eq. [63] into Eq. [20a],

$$v_{\rm ESE}(2\tau) = \exp[-\tilde{g}^2\beta^2\hbar^{-1}CT_1J_{\rm B}(p, \tau/T_1)],$$ [64]

where

$$J_{\rm B}(p, \tau/T_1) = \tilde{g}^{-2}\beta^{-2}\hbar T_1^{-1} \int d^3{\bf r}(1 - v_{\rm B}(2\tau|{\bf r})).$$ [65]

The function $J_{\rm B}$ depends on two dimensionless parameters: τ/T_1 and the degree of
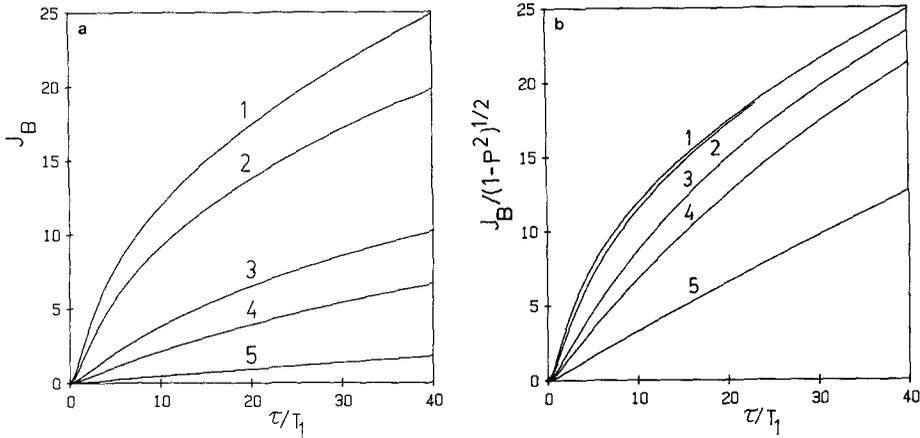
FIG. 6. Time evolution of J_B (a) and the reduced $J_B/(1 - p^2)^{1/2}$ (b) at various degrees of B spin polarization: (1) $p = 0$, (2) $p = 0.6$, (3) $p = 0.9$, (4) $p = 0.95$, (5) $p = 0.99$.

B polarization, p. At $p \rightarrow 0$, Eqs. [64] and [65] coincide with the results of theory of phase relaxation by the spectral diffusion mechanism obtained in the high-temperature limit (see, e.g. (11)). At $T_1 \rightarrow \infty$, $J_B \rightarrow 0$, at $T_1 \rightarrow 0$ Eq. [64] turns into Eq. [61]. At a complete spin polarization ($p \rightarrow 1$), $J_B \rightarrow 0$. At intermediate polarizations of B spins ($0 < p < 1$), J_B is calculated numerically (see Fig. 6a). Analysis of the curves shows that at moderate spin polarizations, when $p \lesssim 0.5$, the following approximate relation holds at any τ/T_1 (see Fig. 6b):

$$J_B(p, \tau/T_1) \approx (1 - p^2)^{1/2} J_B(p = 0, \tau/T_1). \qquad [66]$$

As $\tau/T_1 \rightarrow \infty$ asymptotic relation [66] must hold at any p; however, this approximation is achieved the later, the higher the spin polarizations (see Fig. 6b). In general, as expected, the polarization of B spins reduces their contribution to the ESE signal decay.

Let us consider a general case of spin excitation by microwave pulses. The contribution from dipole–dipole interactions to the ESE signal decay is described by the equation

$$v_{ESE}(2\tau) = \exp\left\{-\tilde{g}^2\beta^2\hbar^{-1}CT_1\left[J_B + \left\langle \sin^2\frac{\theta^{(2)}}{2}\right\rangle_g J_{A0}\right.\right.$$

$$\left.\left. + \left\langle \sin^2\frac{\theta^{(2)}}{2} + 2\sin^2\frac{\theta^{(1)}}{2}\cos^2\frac{\theta^{(2)}}{2}\right\rangle_g J_{A1}\right]\right\}, \qquad [67]$$

where

$$J_{A0} = (1/2)\tilde{g}^{-2}\beta^{-2}\hbar\, T_1^{-1}\exp(-\tau/T_1)$$

$$\times \int d^3\mathbf{r}[A^2/2(R_+^2 + R_-^2)](\cosh(2R_+\tau) - \cos(2R_-\tau)), \qquad [68]$$
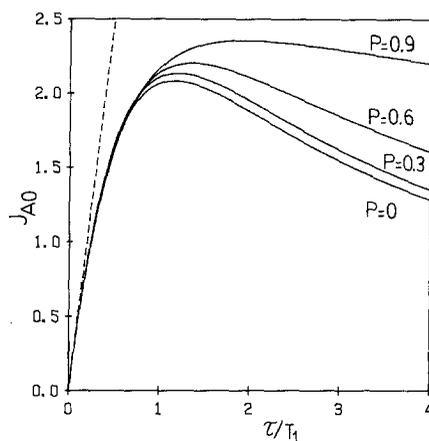
FIG. 7. $J_{AO}(\tau/T_1)$ at various polarization degrees. Dashed line is the asymptote thereto all curves tend at $\tau/T_1 \to 0$, it corresponds to the static limit [59].

$$J_{A1} = (p/2)\tilde{g}^{-2}\beta^{-2}\hbar\, T_1^{-1} \exp(-\tau/T_1)$$

$$\times \int d^3\mathbf{r}[A/(R_+^2 + R_-^2)](R_- \sinh\,(2R_+\tau) - R_+ \sin\,(2R_-\tau)). \quad [69]$$

Here $\langle \cdots \rangle_g$ means averaging over the rotation angles of spins with different resonance frequencies (9–11).

Numerical calculations of $\langle \sin^2\,(\theta/2) \rangle_g$ are reported in Ref. (11). In extreme cases, Eq. [67] is much simpler. In the static limit ($W = 0$), $J_B = J_{A1} = 0$ and Eq. [67] reduces to Eq. [59]. In the high-temperature limit J_{A1} becomes zero and J_{AO} coincides with J_A derived in phase relaxation theory at high temperatures (11). In the limit of fast spin flips J_{AO}, $J_{A1} \sim (T_1/\tau)J_B$ and J_{AO}, J_{A1} can be neglected compared to J_B.

Figures 7 and 8 demonstrate time evolution of J_{AO} and J_{A1} for various polarization
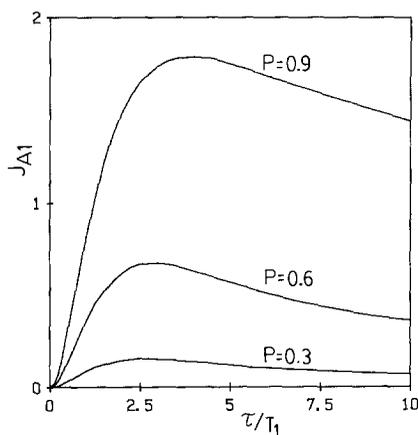
FIG. 8. $J_{A1}(\tau/T_1)$ at various spin polarization degrees.

degrees. It is seen that J_{A0} and J_{A1} pass through a maximum with increasing τ/T_1, and equal zero at $\tau/T_1 \to \infty$. The maximum values of these functions increase with the spin polarization, at $p \to 1$, $(J_{A0})_{max} = (J_{A1})_{max} = 4\pi^2/9(3)^{1/2}$. Note that at $p \to 1$, $J_B \to 0$ and the ESE signal decay is contributed to mainly by J_{A0} and J_{A1}.

CONCLUSION

A very interesting experiment concerned with measuring the contribution from dipole–dipole interactions to ESE signal decays at a high degree of spin polarization has been proposed and successfully realized (*12, 13*). A system of two paramagnetic center species with substantially different g values was studied, namely, Yb^{3+} ($g_{\parallel} = 1.054$) and Tb^{3+} ($g_{\parallel} = 17.777$) ions. The decay of the ESE signal from ions with a lower g value (Yb^{3+} in the study cited) was measured, and the contribution from the dipole–dipole interaction with particles with a higher g value (Tb^{3+}) was analyzed. In the case of Tb^{3+} in the range of helium temperatures, $g\beta H_0 \gtrsim kT$, and the spins are highly polarized. It has been shown (*12, 13*) that polarizations of Tb^{3+} spins reduce the contribution to Yb^{3+} spin phase relaxation by the mechanism of spectral diffusion induced by a random modulation of dipole–dipole interactions between Tb^{3+} and Yb^{3+} due to random flips of Tb^{3+} spins. The method proposed (*12, 13*) opens the way for experimental investigations of effects of dipole–dipole interaction between paramagnetic centers at high-spin polarizations.

Considerable scope for studying spin polarization effects upon ESR spectra shape is offered by construction of spectrometers with the operating magnetic field strength of some 5×10^4 G (2-mm wavelength) (*24*). In such fields spin polarizations are significant at helium temperatures even for paramagnetic particles with g values equal to 2. The experimental results cited (*12, 13*) and the above theoretical considerations show spin polarizations to affect substantially the process of phase relaxation induced by dipole–dipole interactions. Unlike the high-temperature situation, at low temperatures random spin flips do not average the dipole ESR line broadening to zero (see Eqs. [53] and [58]). At the same time, random spin flips average to zero the contribution from instantaneous diffusion to the ESE signal decay (see Eq. [61]) in the same manner as the high-temperature limit. Spin polarizations reduce the contribution of dipole–dipole interactions to the irreversible spin dephasing by the spectral diffusion mechanism (see Figs. 6 and 7). In the general case of spin polarization, the ESE signal decay kinetics cannot be obtained from the corresponding kinetics in the high-temperature limit by a simple reduction of parameters. Altshuler *et al.* (*13*) have made an attempt to realize this reduction. However, this procedure can be applied only to description of the ESE signal decay limits at $\tau/T_1 \to \infty$ and the ESE decay in systems with low polarizations (see Eq. [66]).

In the present work we considered a comparatively simple situation: a center with $S = 1/2$ under the assumption that spin–lattice relaxation is the basic mechanism of random spin flips. In the future it would be desirable to consider particles with arbitrary spins and to investigate mutual spin flips.

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