Weak distortions of the magnetic structure of iron borate

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An investigation has been made of the free induction signal of ^{57}Fe nuclei in iron borate, characterized by an anomalously narrow NMR line at 77 K. The results obtained are explained on the assumption that the hyperfine interaction is anisotropic due to a tilt of the sublattice magnetizations out of the basal plane and that the NMR signal is due to the domain walls.

I. INTRODUCTION

There have been quite a few investigations of the compound $FeBO_3$ in the rf frequency range.[1-9] This is of interest for the following reasons. Firstly, $FeBO_3$ belongs to easy-plane antiferromagnets and such magnetic materials have a number of unusual magnetic properties (weak ferromagnetism, exchange enhancement, etc.). Secondly, this compound is characterized by a fairly large magnetostriction constant, which gives rise to significant magnetoelastic effects on the magnetic properties of this material. Thirdly, the smallest NMR line widths have been observed for $FeBO_3$, with makes it possible to use NMR methods to study very fine effects undetectable by these means in the case of other substances. These are the reasons for the unusual variety of the effects exhibited by $FeBO_3$ in the rf range. The explanation of these effects requires introduction of new concepts and going beyond the usual approximations adoptedin the theory of NMR in magnetic materials.

The magnetic structure of $FeBO_3$ is shown in Fig. 1. Below the Néel temperature ($T_N = 348$ K) the magnetic moments of the Fe^{3+} ions are antiferromagnetically ordered and are slightly tilted relative to the c_3 trigonal axis. The angle of tilt of the magnetic sublattices is 56′ and practically independent of temperature. The effective magnetic exchange, ferromagnetic, and anisotropy fields lie within the easy plane and have the following intensities: $H_E \approx 2.6 \times 10^6$ Oe, $H_D = 85 \pm 7$ kOe, $H_a < 1$ Oe.

We report the results of experimental investigations of a sample of $FeBO_3$ with a narrow NMR line, whose width $\delta\nu$ was only ~ 3 kHz under certain (identified below) conditions. At the NMR frequency of $\nu_n = 75$ MHz the ratio of the width to the line frequency was $\delta\nu/\nu \approx 3 \cdot 10^{-5}$, i.e., it was possible to detect changes in the fields at the nuclei amounting to several oersted and also tilts of the magnetic sublattices of the order of a few angular minutes.

II. SAMPLES AND EXPERIMENTAL METHOD

Our experiments were carried out on $FeBO_3$ single crystals with a natural abundance of the ^{57}Fe isotope. We used samples in the form of rectangular paralellepipeds with dimensions $7 \times 1 \times 1.5$ mm and $4 \times 2 \times 2$ mm. The large face of each sample was parallel to the (111) easy magnetization plane.

We investigated signals representing free induction decay (FID) and its frequency spectrum after Fourier transformation as a function of the amplitude H_1 and duration τ_p of an rf pulse, of the intensity and direction of an external magnetic field $\mathbf{H}_0$, and of the attenuation α_n of ultrasonic pulses transmitted by a sample at the NMR frequency of ^{57}Fe.

We investigated the FID signals using a Bruker CXP-100 pulsed spectrometer operating at 75.395 MHz. The power of rf pulses (P_0) was set at 5×10^2 W and was reduced by an external precision attenuator to -70 dB in steps of 1 dB. The duration of the exciting pulses ranged from 0.5 to 20 μs. The FID signal was stored by sampling for intervals of duration 0.1 and 0.5 μs. We selected a configuration in which the vectors $\mathbf{H}_0$ and $\mathbf{H}_1$ were in the easy magnetic anisotropy plane and were parallel to one another. The main attention was concentrated in the range of weak magnetic fields ($0 \leqslant H_0 \leqslant 200$ Oe), because in this range both the domains and their walls contributed to the NMR signal.

An investigation of the transmission of ultrasonic waves across a sample was made using an acoustic pulsed spectrometer. The propagation wave vector $\mathbf{k}$ of the ultrasound was perpendicular to the (111) basal plane of the crystal and the polarization vector $\boldsymbol{\varepsilon}$ of the ultrasonic waves was in the (111) plane, either perpendicular or parallel to the $\mathbf{H}_0$ external field. The duration of the exciting acoustic pulses was 0.5–2 μs and the repetition frequency was between 500 Hz and 2 kHz. The investigations were carried out at 77 K.

III. EXPERIMENTAL RESULTS

The distinguishing characteristic of our results is detection of two regions of different behavior of the NMR signal, depending on the power of the rf field pulses.

At low powers (when P_0 was attenuated by between -70 and -60 dB and the pulse duration was $\tau_p = 2$ μs) the spectrum of the FID signal in zero magnetic field represented superposition of two wide (~ 30 kHz) lines split by

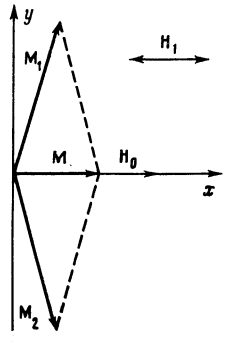

FIG. 1. Magnetic structure of $FeBO_3$ and the polarization of an applied oscillatory field H_1.

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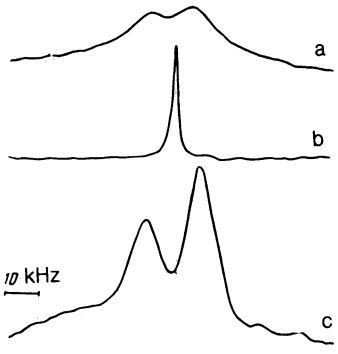

FIG. 2. Spectra of NMR in $FeBO_3$ when $\mathbf{H}_0 \| \mathbf{H}_1 \| (111)$: a) $\Delta P = -70$ dB, $H_0 = 0$; b) $\Delta P = -55$ dB, $H_0 = 0$; c) $\Delta P = -30$ dB, $H_0 = 120$ Oe (ΔP is the power attenuation).

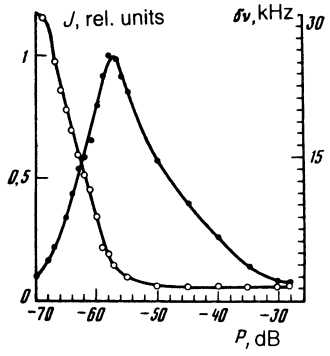

FIG. 4. Dependence of the intensity J(●) and of the width $\delta\nu$(○) of an NMR line on the power P of an rf pulse in the case when $H_0 = 0$.

~20 kHz and differing in the intensity (Fig. 2a). An increase in the power caused these lines to approach one another and reduced their width. In the case of attenuation by between -60 and -55 dB the lines collapsed to form a single narrow line of width $\delta\nu = 3$–4 kHz (Figs. 2b and 3). An increase in the power caused an increase in the NMR signal intensity (Fig. 4).

A further increase in the power (so that the attenuation was less than -50 dB) reduced the intensity of the resonance line, but its width remained constant (Fig. 4). It should be pointed out that the characteristics of the behavior of the NMR signal described above were observed also when the rf pulse duration was increased from 0.5 to 20 μs keeping the power constant (Fig. 3).

At low rf pulse intensities (corresponding to the attenuation by between -70 and -60 dB) we found that both NMR signals decreased rapidly to zero when H_0 was increased to 45–75 Oe (Fig. 5), but the separation between the lines remained unaffected within the limits of experimental error (~10%). It should be stressed that when the NMR signal was excited in this way there was no splitting of any of the lines in the magnetic field H_0.

In the intermediate range of powers (corresponding to attenuation by between -60 and -50 dB) a single NMR line was not split by the application of the field H_0. However, on increase in the power (corresponding to attenuation by $\leqslant -50$ dB) this narrow NMR line began to split in fields of just ~10–20 Oe (Fig. 2c) and in the range $H_0 \geqslant 20$ Oe the splitting rose proportionally to the applied field $\mathbf{H}_0 \| \mathbf{H}_1$ (Fig. 6).

We observed a change in the NMR resonance frequency corresponding to the splitting of the NMR lines in zero magnetic field when a sample was rotated in the (111) plane relative to the direction of the vector $\mathbf{H}_0$ and the intensity was in the range $H_0 \geqslant 100$ Oe.

In ultrasonic experiments we investigated the absorption of the energy of transverse ultrasonic wave pulses as a function of the intensity and direction of the field $\mathbf{H}_0$ in the (111) plane. The orientation of the propagation vector $\mathbf{k}$ and polarization vector $\boldsymbol{\varepsilon}$ of these ultrasonic waves relative to the crystallographic axes was kept constant. In the geometry characterized by $\mathbf{k} \| [111]$ and $\boldsymbol{\varepsilon} \| (111)$ we detected a considerable change in the absorption of ultrasound in fields $H_0 \leqslant 200$ Oe, depending on the orientation of the field $\mathbf{H}_0$ relative to the [001] axis. It follows from Fig. 7 that alternation of the absorption maxima and minima occurs in intervals of 60° and is governed, as shown below, by anisotropic fourth-order terms in the free energy expansion.

IV. INTERPRETATION OF RESULTS

We shall show below that the observed behavior of the NMR signals can be explained by linking them to domain walls. Such a model can explain the splitting of the NMR spectrum, the behavior of signals in the magnetic field, and their dependence on the rf pulse power.

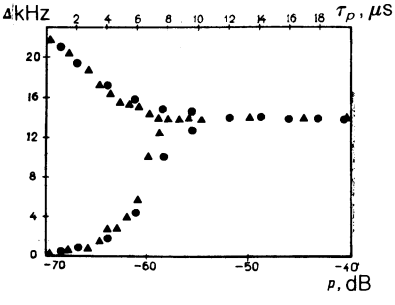

FIG. 3. Splitting of the NMR spectrum shown as a function of the power P(▲) or duration τ_p (●) of an rf pulse when $H_0 = 0$.

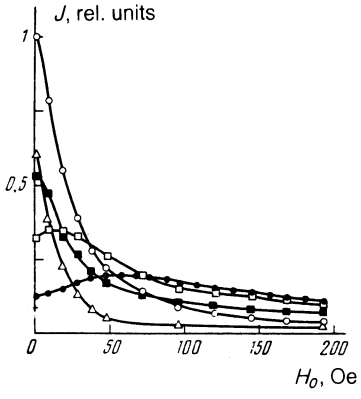

FIG. 5. Dependence of the intensity of the NMR signal on an external field H_0 when $\Delta P = -10$ dB (●), -20 dB (□), -30 dB (■), -40 dB (○), -70 dB (△).

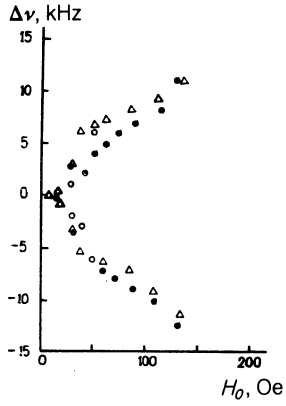

FIG. 6. Splitting of the NMR line in an external magnetic field H_0 when $\Delta P = -10$ dB (●), -20 dB (△), -30 dB (○).

1. Spectrum of NMR frequencies of $FeBO_3$

The splitting of the NMR spectrum at low powers P in zero external field ($H_0 = 0$) can be due to a dynamic frequency shift or an anisotropy of the hyperfine interaction.

The dynamic frequency shift may result in splitting in the NMR spectrum if this splitting is different for different nuclear spins. This splitting mechanism is easily identified on the basis of the characteristic temperature dependence $\delta\nu_p = \propto m \propto T^{-1}$ reported in Ref. 10 (m is the nuclear magnetization with its dependence on the temperature T governed by the Curie law). In view of this dependence the splitting at $T = 4.2$ K ought to exceed the splitting at $T = 77$ K by a factor of 18, whereas the opposite relationship has been observed experimentally.[3,8] Moreover, estimates of $\delta\nu_p$ from a familiar theoretical expression describing an easy-plane antiferromagnet[10] with parameters corresponding to $FeBO_3$ at $T = 77$ K gave a value $\delta\nu_p \approx 1$ kHz, which was considerably less than the observed splitting, amounting to 20 kHz. Therefore, the influence of the dynamic frequency shift on the NMR spectrum can be ignored.

It remains to consider the influence of the hyperfine interaction anisotropy. We shall begin with the familiar expression for the NMR frequency[10]

$$\omega_{nj} = \gamma_n |\mathbf{H}_{nj} + \mathbf{H}_0|, \tag{1}$$

where γ_n is the gyromagnetic ratio, H_0 is the external magnetic field

$$H_{nj}{}^{\alpha} = \sum_{\beta j'} A_{jj'}^{\alpha\beta} M_{j'}{}^{\beta}, \tag{2}$$

α is the component of the hyperfine field at the nucleus of an atom in a sublattice j ($j = 1$ or 2), $A_{jj'}^{\alpha\beta}$ are the components of the hyperfine interaction tensor, and M_j, is the magnetization of the sublattice j'. The symmetry of the $A^{\alpha\beta}$ tensor is determined by the environment of the atom. In the case of the iron atoms in $FeBO_3$ allowing for the smallness of the components of the hyperfine interaction tensor described by $|A_{jj}^{\alpha\alpha} - A_{jj}^{\beta\beta}|$, $|A_{jj}^{\alpha\beta}| \ll |A_{jj}^{\alpha\alpha}|$ when $\alpha \neq \beta$ and by the inequality $|A_{jj'}^{\alpha\alpha}|$ when $j \neq j'$, we obtain the following expression for ω_{nj}:

$$\omega_{nj} = \omega_n + \delta\omega_n \Delta\theta^2 - (-1)^j \gamma_n H_0 \cos\varphi_H, \tag{3}$$

where

$$\omega_n = \gamma_n A_1 M_0, \quad \delta\omega_n = \gamma_n M_0 (A_2 - A_1), \quad A_1 = |A_{jj}^{xx}| = |A_{jj}^{yy}|, \quad A_2 = |A_{jj}^{zz}|, \tag{4}$$

$\Delta\theta$ is the angle between the sublattice magnetization M_j and the (x,y) plane [which is the (111) basal plane of the crystal], z is the sixfold ([111]) axis, M_0 is the sublattice magnetization, φ_H is the angle between the field $\mathbf{H}_0$ and the antiferromagnetic vector satisfies $L = M_1 - M_2$, where $\mathbf{M} = (\mathbf{M}_1 + \mathbf{M}_2) \perp \mathbf{L}$.

It follows from Eq. (3) that if $\Delta\theta = 0$ and $H_0 = 0$, the frequencies ω_{nj} are independent of the sublattice orientations in the basal plane. In this case the NMR spectrum consists of a single line (which may be broadened by defects distributed at random in a crystal). Hence it follows that the splitting observed when $H_0 = 0$ (Fig. 2a) can only be due to a tilt of the vectors $\mathbf{M}_{1,2}$ out of the basal plane. One of the reasons for this tilt is the fourth-order anisotropy.

In fact, in this approximation the magnetic anisotropy energy of an $FeBO_3$ crystal is given by[12]:

$$H_{an} = {}^1/_2\, bL_z^2 + {}^1/_2\, g[(L_x + iL_y)^3 + (L_x - iL_y)^3] L_z. \tag{5}$$

The angle between L and the basal plane xy is found from the condition for a minimum of (5) which, allowing for $L^2 = 4M_0^2$, gives

$$\Delta\theta(\varphi) = (2g/b) M_0^2 \cos 3\varphi = \Delta\theta_0 \cos 3\varphi. \tag{6}$$

where φ is the azimuthal angle of L in the (x,y) plane.

One of the experimental proofs that the $\mathbf{M}_1$ and $\mathbf{M}_2$ vectors are tilted out of the basal plane has been obtained by investigating acoustic NMR. According to Ref. 10, if the magnetic moments of the sublattices are not tilted out of the basal plane, the absorption of the energy of transverse acoustic waves due to the magnetoelastic interaction should vanish when the wave vector $\mathbf{k}$ is perpendicular to the (111) basal plane.[1)] It follows from Fig. 7 that the angular dependence of the absorption by the wave with the vector $\mathbf{k} \| [111]$ as a function of the orientation of the vector $\mathbf{L}$ in the (111) plane has a symmetry of the form $\cos 3\varphi \cos 2\varphi$. The factor $\cos 3\varphi$ is related to the angular dependence $\Delta\theta(\varphi)$ given by Eq. (6) and $\cos 2\varphi$ is due to the dependence of the absorption on the angle between the acoustic wave polarization and the vector $\mathbf{L}$ (Ref. 10).

A more rigorous confirmation of the tilt on the sublat-

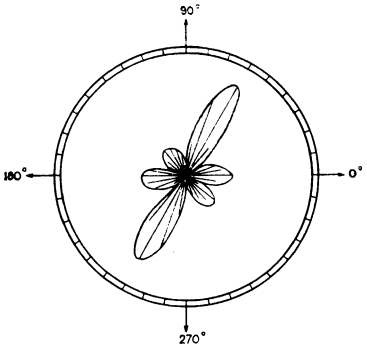

FIG. 7. Angular dependence of the intensity of a transverse ultrasonic pulse in the case when $\mathbf{k} \| [111]$ and $\mathbf{H}_0$ is rotated in the (111) plane.

tice magnetization vectors was provided by the observed change in the resonance frequency when a sample was rotated in the spectrometer coil about the [111] axis relative to the vector $\mathbf{H}_0$. The nature of the frequency shift agreed with the anisotropy of the absorption of ultrasound. The dependence of the NMR frequency ω_{nj} on the sublattice orientation in the basal plane, characterized by a period $\pi/3$, can account for the splitting of the NMR spectrum

$$\Delta\omega = \delta\omega_n \Delta\theta_0^2,$$

where such splitting (as shown below) may be associated both with domains and with their walls.

It should be pointed out that a tilt of the sublattice magnetic moments out of the basal plane had been reported earlier for the antiferromagnets $CoCO_3$ and $NiCO_3$ (Ref. 11).

2. Spectrum of NMR signals due to domains and their walls

We shall consider the specific case when $\delta\omega_n > 0$ in Eq. (3). Then if $H_0 = 0$, the lf signal is associated with domains in which the orientation of L in the basal plane is governed by the condition $\cos^2 3\varphi = 0$, whereas the hf signal is associated with the domains characterized by $\cos^2 3\varphi = 1$. We shall now consider how the signals behave in the presence of a static magnetic field H_0 when an oscilatory field $\mathbf{H}_1$ has the following two orientations; $\mathbf{H}_1 \| \mathbf{H}_0$ and $\mathbf{H}_1 \perp \mathbf{H}_0$.

If $\mathbf{H}_1 \| \mathbf{H}_0$, we have to allow for the effects of two types. The first is that the field H_0 tends to align the vectors $\mathbf{M}$ along $\mathbf{H}_0$ in all the domains. The second effect is a dependence of the gain of the NMR signals on the angle between $\mathbf{H}_1$ and $\mathbf{M}$. Since in the case when $\mathbf{H}_1 \| \mathbf{M}$ the gain approaches zero, $\eta \to 0$, it follows that an increase in H_0 should cause the hf and lf signals to disappear. We can see from Fig. 5 that this is not in conflict with the experimental results.

If $\mathbf{H}_1 \perp \mathbf{H}_0$, then in the case of domains characterized by $\mathbf{M} \| \mathbf{H}_0$, the polarization of the external oscillatory field is characterized by $\mathbf{H}_1 \perp \mathbf{M}$, which corresponds to the maximum of the gain.[10] It therefore follows that one of the signals should remain when H_0 increases, but its spectrum may not be split by the field H_0. This follows from Eq. (3), because if $\mathbf{H}_0 \| \mathbf{M}$, we have $\mathbf{H}_0 \perp \mathbf{L}$, i.e., $\cos\varphi_H = 0$.

The possibility that fine structure the NMR spectrum may appear due to the nuclei in domain walls of antiferromagnets as a result of the hyperfine interaction anisotropy was considered in Ref. 14. It is due to the fact that the parts of the domain walls where the orientations of the $\mathbf{M}_1$ and $\mathbf{M}_2$ sublattices correspond to the maximum and minimum values of the frequency ω_{nj} in Eq. (3) can make the greatest contribution to the NMR signal intensity. We shall use this approach to analyze the behavior of the signal due to domain walls in a field H_0 when $\mathbf{H}_1 \| \mathbf{H}_0$ and $\mathbf{H}_1 \perp \mathbf{H}_0$.

In the case when $\mathbf{H}_1 \| \mathbf{H}_0$ both static and alternating fields act on the same walls. Since a sufficiently strong field H_0 destroys such walls, the NMR signal should disappear as H_0 increases, which is again in agreement with the experimental results (Fig. 5). Moreover, since displacements of domain walls create a demagnetizing field, which compensates the field H_0 inside the sample, it follows that H_0 cannot penetrate the sample until the displacement processes are complete. This means that this field cannot influence the splitting of the NMR spectrum.

If the field orientations are such that $\mathbf{H}_0 \perp \mathbf{H}_1$, we can speak of two types of domain wall. The first includes the walls which are displaced by the field H_1 and, consequently, are responsible for the NMR signal. The walls of the second type are displaced only by the field H_0, whereas H_1 does not act on them and therefore they make no contribution to the NMR signal. The walls displaced by both fields will be ignored here, because they behave in the same way as in the $\mathbf{H}_0 \| \mathbf{H}_1$ case discussed above. The influence of the field H_0 begins at relatively high values, when the processes of rotation of the magnetization vectors in the domains are activated.

Since the angular anisotropy in the absorption of ultrasound and in the change in the resonance frequency can be observed in fields below 200 Oe, this can be regarded as a confirmation that the main contribution to the splitting in $H_0 = 0$ comes from domain walls.

However, the above analysis cannot account unambiguously for the splitting of the NMR line in the field H_0 when $\mathbf{H}_1 \| \mathbf{H}_0$. Clearly, such splitting is due to oscillations of $\mathbf{M}$ in domains with a complex structure. In this case the field $\mathbf{H}_0$ is parallel to $\mathbf{H}_n$ for one sublattice and antiparallel to $\mathbf{H}_n$ for the other sublattice [Eq. (3)].

3. Dependence of the spectrum of the NMR signal on the power of exciting pulses

One of the mechanisms that ensure such a dependence is related to saturation of the free precession signal resulting from inhomogeneities of the gain. We can describe this effect using an expression for the free precession signal intensity after an rf pulse of duration τ_p (Ref. 15):

$$J(t) = \int dr\, \eta(r)\, m_0 \sin[\theta(r)]\, e^{i\varphi(r)} e^{i\omega_n(r)(t-\tau_p)} \qquad (7)$$

where r are the coordinates of the points at which the signals are generated; $\eta(r)$ is the gain at such a point; m_0 is the equilibrium nuclear magnetization, the spatial dependence of which will be ignored; $\theta(r)$ and $\varphi(r)$ are the polar and azimuthal angles of $\mathbf{m}$ after a pulse; $\omega_n(r)$ is the NMR frequency.

We shall now allow for the fact that the spatial dependences $\eta(r)$, $\theta(r)$, $\varphi(r)$, and $\omega_n(r)$ are governed by the position of an excited nuclear spin in a domain wall, so that it is more convenient to use $\omega_n(r)$ as the integration variable in place of r. This allows us to rewrite Eq. (7) in the form

$$J(t) = m_0 \int d\omega_n\, G(\omega_n)\, \eta(\omega_n) \sin[\theta(\omega_n)]\, e^{i\varphi(\omega_n)} e^{i\omega_n(t-\tau_p)} \qquad (8)$$

where $G(\omega_n)$ is the distribution function of the frequencies of nuclear spins, which is determined by the domain wall structure and the angular dependence of the NMR frequency.[14] In the absence of any other inhomogeneities the signal amplitude considered as a function of the frequency ω_n (obtained from a spectrum analyzer) should be described by

$$J(\omega_n) = m_0 \eta(\omega_n) \sin[\theta(\omega_n)] G(\omega_n). \qquad (9)$$

In particular, the splitting observed in the NMR spectrum for $H_0 = 0$ at low powers P (Fig. 2a) is most probably due to certain features of $G(\omega_n)$ at the maximum and minimum values of the frequency ω_{nj} of Eq. (3). We shall show that an increase in P enhances the importance of the scatter of the domain walls with respect to their mobilities, dimensions, and other properties which affect the gain η. We can account

for the influence of this scatter by averaging Eq. (9) over the scatter:

$$\overline{J(\omega_n)} = m_0 G(\omega_n)\,\overline{\eta(\omega_n)\sin[\theta(\omega_n)]}$$

$$= m_0 G(\omega_n)\int d\eta\, g(\eta)\,\eta \sin[\theta(\eta)], \qquad (10)$$

where $g(\eta) \equiv g(\eta(\omega_n))$ is the function representing the distribution of the gain η on the scale of the frequency ω_n (i.e., the distribution function at a given point in a domain wall).

The properties of the integral on the right-hand side of Eq. (10) will be discussed by considering a resonance pulse of frequency ω which is identical with ω_n. In this case the angle of rotation **m** under the influence of an rf pulse of duration τ_p is given by the expression[15]

$$\theta(\eta) = \gamma_n H_1 \eta \tau_p \qquad (11)$$

and for a Gaussian distribution function

$$g(\eta) = (2/\pi)^{1/2}(\Delta\eta)^{-1}\exp\{-(\eta-\eta_0)^2/2\Delta\eta^2\}$$

when the width of the scatter is large so that $\Delta\eta \gg \eta_0$, we obtain

$$\overline{J(\omega_n)} = m_0 G(\omega_n)\Delta\eta(\omega_n)[\gamma_n \tau_p \mathrm{H}_1 \Delta\eta(\omega_n)]$$
$$\times \exp\{-[\gamma_n \tau_p \mathrm{H}_1 \Delta\eta(\omega_n)]^2/2\}. \qquad (12)$$

It follows from Eq. (12) that if $\gamma_n H_1 \Delta\eta\tau_p < 1$, the amplitude of the signal increases with increasing in H_1 and τ_p, but if $\gamma_n H_1 \Delta\eta\tau_p > 1$, it falls exponentially (the signal saturation effect displayed in Fig. 4). If we assume that the scatter of $\Delta\eta$ is greater where the gain $\eta(\omega_n)$ is larger, then the signal due to the nuclear spin for large values of $\eta(\omega_n)$ is the first to be saturated, i.e., saturation occurs at the centers of domain walls. It therefore follows that at sufficiently high powers of the rf pulse the NMR signal should be generated by the edges of the domain walls where the values of the gain $\eta(\omega_n)$ are minimal.

The above discussion demonstrates that the observed splitting of the NMR spectrum in $H_0 = 0$ is due to the hyperfine interaction anisotropy which in turn appears because the sublattice magnetization vectors are tilted out of the basal plane. This appearance of the splitting in $H_0 = 0$ and the line narrowing line as the rf pulse power increases occur because of inhomogeneous saturation of the NMR signal in domains and their walls. The splitting of the NMR spectrum in a magnetic field when $\mathbf{H}_1 \| \mathbf{H}_0$ is due to the different signs of $H_0 \cos\varphi_H$ in domains of the two sublattices.

[1)]This conclusion cannot be regarded as final if we use the results of Ref. 13 where it is shown that the magnetoelastic energy includes terms of the $2B_{14}[(L_x^2 - L_y^2)U_{yz} + 2L_x L_y U_{xz}]$ type, which ensure the absorption of transverse sound propagating along the [111] axis.

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