Effect of paramagnetic impurity on spin-spin and spin-lattice relaxation of ^{57}Fe nuclei in hematite

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The effect of paramagnetic impurities on spin-spin and spin-lattice relaxation of nuclei is studied experimentally and theoretically in hematite containing 0.8 wt.% Sn^{4+}. The absence of a Morin transition from such crystals and enrichment with ^{57}Fe to 85% made possible measurement, by the spin-echo method, of T_1 and T_2 in the large temperature range 4.2–400 K. Broad maxima of $1/T_1$ and $1/T_2$ were observed in the region of 70 K. It is shown that at $T<200$ K the decisive factor is the interaction of the nuclei with the paramagnetic Fe^{2+} impurities that are produced in equal amounts, by virtue of the electroneutrality condition, when the Sn^{4+} is introduced. The observed contribution to $1/T_1$ and $1/T_2$ with a characteristic maximum in the "low-temperature" region is interpreted within the framework of the impurity relaxation absorption (the slow relaxation mechanism). Analysis of the experimental data yielded the frequency of the impurity local mode ($\omega_0 \approx 10^{11}$ sec) and identified the mechanisms of the spin-lattice relaxation of the Fe^{2+} impurity. At $T>90$ K the hopping mechanism of relaxation predominates, $\tau_1^{-1}(T)=\tau_{1\infty}^{-1}\exp(-E/T)$, $\tau_{1\infty}=10^{-11}$ sec, $E=1500$ K; at $T<80$ K the two-magnon relaxation mechanism with $\tau_1^{-1}(T)=AT^n$, $n=3$ is the principal one. The substantial difference between T_1 and T_2 at $T<80$ K indicates that the correlation effects in the subsystem of the enriched ^{57}Fe nuclei play an important role in this low-temperature region.

INTRODUCTION

Investigations of nuclear relaxation in yttrium garnet[1,2] as well in certain spinel ferrites (manganese ferrite[3] and magnetite[4]) have shown that the presence in them of paramagnetic rare-earth or transition-element impurities, e.g., Fe^{2+} ions, leads to the appearance of a low-temperature, maximum of the rates of the spin-lattice[1–4] and spin-spin[4] relaxations.

This behavior of the relaxation frequency T_1^{-1} was attributed to a manifestation of a slow-relaxation mechanism,[5–7] viz., the perturbation of the thermal distribution in the system of the impurity energy levels under the influence of the NMR signal, followed by their relaxation in the thermostat. The rate of the spin-lattice nuclear relaxation T_1^{-1} was expressed in Ref. 3 in terms of the linewidth of the FMR with the NMR frequency which is not at resonance with the electron subsystem. This phenomenological hypothesis was based on the fact that in magnetically ordered systems the nuclear and electronic magnetizations are dynamically interrelated, suggesting correlation in the nuclear subsystems. In Refs. 1, 2, and 4, on the contrary, the nuclear magnetic relaxation was considered under conditions of total absence of correlation in the nuclear subsystem, when the relation $T_2=2T_1$ is valid. The presence of low-temperature maxima of T_1^{-1} and T_2^{-1} was also attributed to the slow-relaxation mechanism. A theoretical analysis of nuclear spin-lattice relaxation in ferrites was carried out in Refs. 6 and 7. The expression obtained then for T_1^{-1} was

$$\frac{1}{T_1}\sim c\frac{\omega_0^2}{\omega_E^{1/2}\varepsilon_0^{1/2}}\frac{\omega_n^2\tau_1}{(\omega_n\tau_1)^2+1},$$

where ε_0 is the FMR frequency, ω_E is the exchange frequency, ω_n is the NMR frequency, c is the impurity density, ω_0 is the frequency of the local mode, and τ_1 is the impurity spin-lattice relaxation time. It is easily seen that the maximum relaxation rate is reached at $\omega_n\tau_1\approx 1$.

It is of interest to study the influence of impurity ions on nuclear-relaxation processes in other types of magnets, particularly in antiferromagnets with easy-plane anisotropy (AFEP) which have weak ferromagnetism, a typical representative being hematite (α-Fe_2O_3). The relaxation times for hematite were measured earlier only at room temperatures and were qualitatively attributed to the mechanism of thermal excitation of Winters magnons.[8]

From the theoretical point of view, investigations of this type are important, inasmuch as in AFEP the coupling of the nuclear and impurity spins is particularly strong, in view of the effect of exchange enhancement of the hyperfine interaction (HFI). We note that experimental investigations of the temperature dependence of the AFMR linewidth[9] and of the relaxation frequency of spin waves (SW) with wave vectors $k\sim 10^5$ cm^{-1} (Ref. 10), for another typical representative, $FeBO_3$, of high-temperature AFEP have revealed the importance of impurity relaxation mechanisms, viz., a low temperature peak was observed in the relaxation rate. A theoretical analysis[11] shows that in the temperature region where the relation $\varepsilon_k\tau_1\approx 1$ is satisfied (ε_k is the SW frequency) the relaxation absorption is the principal relaxation mechanism in the magnetic subsystem at not too high impurity densities.

In impurity-free hematite at $T=T_M\approx 260$ K a transition takes place from the easy-plane to the easy-axis phase. The result is a sharp decrease of the NMR gain and the observation of the NMR becomes difficult. This circumstance prevents investigation of T_1 and T_2 in a wide range of temperatures. This difficulty, however, can be avoided by using

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the fact that T_M depends strongly on the species and density of the impurity atoms. We used in the present study α-Fe_2O_3 crystals enriched with the isotope ^{57}Fe to 85% and containing 0.8 wt.% of Sr^{4+}. Preceding investigations of NMR (Ref. 12) and of antiferromagnetic resonance[13] have shown that these crystals retain a weak ferromagnetism at least down to 1.2 K.

To suppress the influence of the contribution made to the NMR signal by the highly mobile domain walls, the crystals were quenched from 1300 °C (for more details see Ref. 12).

PROCEDURE OF MEASURING T_1 AND T_2

We measured T_1 and T_2 by the spin-echo method using the pulse spectrometers ISSH-1-13 designed by the Special Design Office of the Institute of Radio and Electronics of the USSR Academy of Sciences and SXP-4-100 manufactured by the Brooker Company. T_2 was usually determined by measuring the decrease of the echo amplitude with increasing time interval between the pulse pairs. For a more reliable measurement of T_1 we used various methods made possible by the design features of the employed spectrometers. Depending on the concrete experimental conditions (temperature, values of T_1), T_1 was determined as follows: a) by time restoration of the longitudinal component of the longitudinal component m_z of the nuclear magnetization in a sequence of three pulses; b) by restoring m_z after a series of saturating pulses, c) from the dependence of the echo amplitude on the pulse-pair repetition frequency, d) from the change of the stimulated-echo amplitude.

The optimal spin-echo observation regime, which ensures an exponential decrease of the signal amplitude, was reached using pulses of almost equal duration of the order of 4 μsec and a voltage $\sim$500 V on the measuring coil.

The measurements were made in the temperature range 4.2–400 K using draft cryostats with the temperature electronically stabilized to within $\pm$0.1 K.

THEORY OF NUCLEAR RELAXATION IN AFEP WITH PARAMAGNETIC IMPURITIES

In the study of nuclear magnetic relaxation due to relaxation absorption by paramagnetic impurities we begin with a Hamiltonian in the form

$$\hat{\mathcal{H}}=\hat{\mathcal{H}}_0+\hat{\mathcal{H}}_{n-imp}+\hat{V}, \tag{1}$$

where

$$\hat{\mathcal{H}}_0=-\omega_n m_0^z-\omega_0\sigma_0^z$$

describes the precession of the nuclear spins in the hyperfine field and the precession of the impurity spins in the molecular field, respectively,

$$\hat{\mathcal{H}}_{n-imp}=-\frac{1}{N}\sum_{\mathbf{k}} V_{\mathbf{k}}^z(m_{\mathbf{k}}^-+m_{-\mathbf{k}}^+)\delta\sigma_{\mathbf{k}}^z \tag{2}$$

describes the interaction of the transverse components of the nuclear spins with the longitudinal component of the paramagnetic impurity, and the operator $\hat{V}$ describes the interaction of the impurities with the thermostat. Here $m_r^\nu=I_g^\nu+I_f^\nu$; $\mathbf{I}_g$ and $\mathbf{I}_f$ are the spins of the nuclei of the two sublattices, $m_{\mathbf{k}}^\nu$ is the Fourier component of $m^\nu(\mathbf{r})$,

$$\sigma_{\mathbf{k}}^\nu=\frac{1}{N}{\sum_{\mathbf{r}_n}}' \sigma^\nu(\mathbf{r}_n)\exp(i\mathbf{k}\mathbf{r}_n),$$

where the summation is over the site occupied by the impurities,

$$V_{\mathbf{k}}^z=\frac{\alpha}{4}\omega_0\frac{\omega_n J_0}{\varepsilon_{\mathbf{k}}^2} \tag{3}$$

is the amplitude of the corresponding interaction, $J_0=\omega_E/S$ is the exchange constant in an ideal matrix, ω_0 is the frequency of the local mode of the impurity as $H\to 0$, ω_n is the frequency of the unshifted NMR, α is a factor of order unity and depends on the mutual orientation of the quantization axes of the electron spins, of the matrix, and of the impurity (if spin-orbit effects are neglected, when the coupling of the impurity spin σ with the matrix spin S is determined by isotropic exchange, $\alpha=0$ and the slow-relaxation mechanism is ineffective),

$$\varepsilon_k=(\varepsilon_0^2+s^2k^2)^{1/2},\qquad \varepsilon_0^2=\mu^2[H(H+H_D)+2H_E H_{mes}]+cBJ_0\omega_0 \tag{4}$$

is the SW spectrum, where s is the SW velocity, H is the external magnetic field, $\mu=g\mu_0$, μ_0 is the Bohr magneton, H_D is the Dzyaloshinskiĭ field, and H_{mes} is the striction field (we use a system of units with $\hbar=1$ and $k_B=1$).

It can be shown[3)] that the time T_1 of the spin-lattice relaxation of the nuclei takes in the relaxation mechanism the form

$$\frac{1}{T_1}=2c\frac{b'}{b}B'\frac{1}{T}\frac{\omega_n\tau_1}{(\omega_n\tau_1)^2+1}v_0\int\frac{d\mathbf{k}}{(2\pi)^3}|V_{\mathbf{k}}^z|^2, \tag{5}$$

where

$$B=\sigma B_\sigma(\sigma\omega_0/T),\quad b=IB_I(I\omega_n/T)$$

are respectively the polarizations of the impurities and of the nuclei, $B'=dB/dy$, B_σ is the Brillouin function, τ_1 is the spin-lattice relaxation time of the paramagnetic impurities, and v_0 is the unit-cell volume. In the high-temperature limit $T\gg\omega_0$ we obtain from (5), taking into account the explicit form of the matrix element $\mathbf{V}_{\mathbf{k}}^z$ for AFEP,

$$\Gamma_1=\frac{1}{T_1}=c\frac{\sigma(\sigma+1)}{24\pi}\frac{\eta^3}{S^2}\beta_1\frac{\omega_0^2}{\omega_E\varepsilon_0}\frac{\omega_n^2\tau_1}{(\omega_n\tau_1)^2+1}. \tag{6}$$

Here $\eta=\omega_E/\Theta_N$ is a structure factor, $\Theta_N=s/v_0^{1/3}$, $\beta_1=\alpha^2$.

The spin-spin relaxation time is given by

$$1/T_2={}^1/_2\Gamma_1+\Gamma_2, \tag{7}$$

where

$$\Gamma_2=\Gamma_2(0),\quad \Gamma_2(k)=bcB'b\frac{1}{T}\frac{\omega_n\tau_1}{(\omega_n\tau_1)^2+1}|V_k^z|^2. \tag{8}$$

The quantity Γ_2 can be expressed in the form

$$\Gamma_2=cB'\frac{I(I+1)}{3S^2}\beta_1\frac{\omega_0^2}{T^2}\frac{\omega_n^2\omega_E^2}{\varepsilon_0^4}\frac{\omega_n^2\tau_1}{(\omega_n\tau_1)^2+1}. \tag{9}$$

The spin-lattice relaxation time τ_1 of the paramagnetic im-

purites in expressions (6) and (9) depends on the time. These expressions contribute therefore to the relaxation frequencies Γ_1 and Γ_2; the contribution has a characteristic maximum at temperatures satisfying the condition $\omega_n \tau_1(T) \approx 1$. At high temperatures τ_1 is determined by the activation mechanism

$$\tau_1^{-1} = \tau_{1\infty}^{-1} \exp(-E/T), \tag{10}$$

and at relatively low temperatures, when the relaxation rate given by (10) is exponentially small, τ_1 is determined by the interaction with the spin waves. At $\omega_0 > \varepsilon_0$ and $c < c^*$ ($c^* \sim (T/\omega_0)^2 \times |\omega_0^2 - \varepsilon_0^2|^{3/2}/\omega_E^3$ is the characteristic density at which the correlation effects in the impurity subsystem of the AFEP become important[14] τ_1 is determined by single-magnon processes:

$$\frac{1}{\tau_1^{1m}} = \frac{\eta^3}{8\pi S} (\omega_0^2 - \varepsilon_0^2)^{1/2} \frac{\omega_0 T}{\omega_E^2}. \tag{11}$$

In the case $\omega_0 < \varepsilon_0$ or $\omega_0 > \varepsilon_0$, $c > c^*$ the relaxation time τ_1 is determined by two-magnon relaxation processes:

$$\frac{1}{\tau_1^{2m}} = \frac{3\zeta(3)}{4\pi^3} \frac{\eta^6}{S^2} \beta_2 \frac{\omega_0^2}{\omega_E} \left(\frac{T}{\omega_E}\right)^3. \tag{12}$$

We have used here the approximation $T \gg \omega_0$. The factor β_2 in (12), just as β_1, depends on the relative orientation of the quantization axes (see below).

RESULTS AND THEIR DISCUSSION

The measured T_1 and T_2 are shown in Fig. 1. The values of $1/T_1$, measured by various methods, are all marked by like circles. The scatter of the points gives an idea of the error in the determination of the relaxation time when various procedures are used.

It can be seen from Fig. 1 that for the spin-lattice relaxation rate $1/T_1$, just as for the spin-spin relaxation rate $1/T_2$,

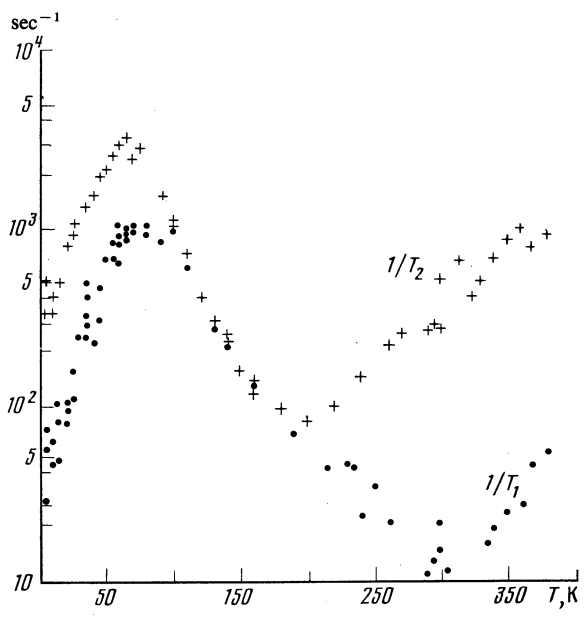

FIG. 1. Temperature dependences of the rates of the longitudinal ($1/T_1$, points) and transverse ($1/T_2$, crosses) of the nuclear relaxation for hematite doped with Sn^{4+}.

deep and broad maxima are observed at $T = T_m \cong 70$ K. An external constant magnetic field applied in the basal plane and certain to make the crystal one-domain (up to 1.5 kOe) does not change, within the limit of errors, the values of T_1 and T_2 or the character of their temperature dependence, so that the anomalies observed cannot be attributed to peculiarities in the dynamics of the domain walls.

As the temperature is lowered or raised from T_m, the relaxation rate decreases significantly (by more than a decade). If is is assumed that the maximum of the relaxation rate is due to relaxational absorption, it can be seen that at temperatures $T < 200$ K the spin-spin and spin-lattice relaxations are due to interaction with the paramagnetic impurity. In our case this impurity constitutes the Fe^{2+} ions that inevitably appear in equal numbers with the Sn^{4+} ions introduced into the hematite, by virtue of the electroneutrality condition.

We consider first T_1. In the temperature region in which the relaxation mexchanism predominates, T_1 is described by Eq. (6), and the condition $\omega_n \tau_1 = 1$ is satisfied at the maximum point. This condition enables us, knowing the experimental value $T_1^{-1} = 10^3$ sec^{-1} at the point of maximum T_m, to determine the frequency ω_0 of the local mode of the impurity. Recognizing that for the given α-Fe_2O_3 sample we have $c \cong 10^{-2}$ and $\omega_n/2\pi = 70$ MHz, and that $\sigma = 2$ for Fe^{2+} ions, we rewrite Eq. (6) at $T = T_m$ in the form

$$\frac{1}{T_1} = 10^{-3} \omega_n \frac{\omega_0^2}{\omega_E \varepsilon_0}, \quad \omega_E \cong 10^{14} \text{ sec}^{-1}, \quad \varepsilon_0 = 10^{11} \text{ sec}^{-1}, \tag{13}$$

from which we obtain directly $\omega_0 \approx 10^{11}$ sec^{-1}. Since the experiments were performed at $T \geqslant 4.2$ K, the assumption $T > \omega_0$ is valid in the entire temperature range. From the experimental $T_1(T)$ dependence we can deduce the temperature dependence of the spin-lattice relaxation time $\tau_1(T)$ of the paramagnetic impurities. From (6) it follows that

$$\omega_n \tau_1^{\pm}(T) = \frac{T_1(T)}{T_1(T_m)} \pm \left[\left(\frac{T_1(T)}{T_1(T_m)}\right)^2 - 1\right]^{1/2}. \tag{14}$$

At $T < T_m$ it is necessary to use the upper sign of the square root, and at $T > T_m$ the lower. The two roots are equal at $T = T_m$.

Figure 2a shows the temperature dependence of $\tau_1(T)$ in the coordinates $\ln \tau_1^{-1}$ and $1/T$. It can be seen from the figure that at relatively high temperatures $T > 80$ K ($T^{-1} < 12 \times 10^{-3}$) the experimental data fit a straight line in accord with Eq. (10). From the slope of this line we obtain the activation energy $E = 1500$ K (0.13 eV). Such an energy is typical of the hopping mechanism and agrees with the experimental data of Refs. 3 and 4. Extrapolation of the data to the region $T \to \infty$ yields the pre-exponential factor $\tau_{1\infty}^{-1} = 10^{11}$ sec^{-1}. The deviation of the experimental points from the straight line in Fig. 2a indicates that Eq. (10) no longer holds at $T < 80$ K. Figure 2b shows a plot of $\tau_1^{-1}(T)$ at $T < 80$ K in a doubly logarithmic scale. It can be seen that the data are described in sufficiently large temperature range by a relation of the form $\tau_1^{-1} = AT^n$, $n \approx 3$, in agreement with Eq. (12). The deviation of the experimental points of Fig. 2b from the straight line at $T \geqslant 80$ K indicates a transition to the Arrhenius law (see Fig. 2a).

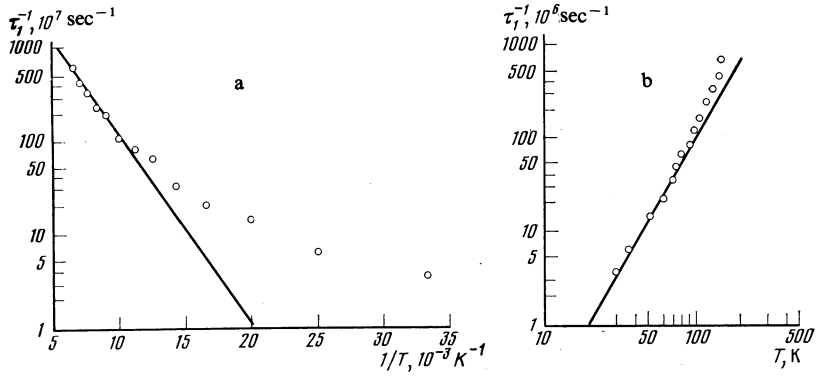

FIG. 2. Temperature dependence of the impurity spin-lattice relaxation time τ_1: a—high-temperature section corresponding to the Arrhenius law; b—low-temperature section corresponding to the power-law dependence $\tau_1^{-1} = AT^n$ ($n \approx 3$).

The analysis of the experimental data on the temperature dependence of T_1^{-1} at $T < 200$ K yields thus the local-mode frequency $\omega_0 = 10^{11}$ sec^{-1} and explains the mechanisms of the spin-lattice relaxation of the paramagnetic impurities. At $T > 80$ K the impurities relax by activation:

$$\tau_1^{-1} = \tau_{1\infty}^{-1} \exp(-E/T) \quad (\tau_{1\infty} = 10^{-11} \text{ sec}, E \cong 1500 \text{ K}),$$

and at $T < 80$ K the spin-lattice relaxation of the impurities is due to the two-magnon relaxation processes (12). The latter fact indicates that the condition $c > c^*$ is satisfied in the crystal investigated ($c \sim 10^{-2}$) and that the correlation in the impurity subsystem is substantial.

We proceed to consider the spin-spin relaxation T_2^{-1}. We can rewrite (7) in the form

$$\frac{1}{T_2} = \frac{1}{T_1}\left(\frac{1}{2} + \Lambda\right), \tag{15}$$

where

$$\Lambda = (T^*/T)^2, \quad T^* \propto \omega_n (\omega_E/\varepsilon_0)^{1/2}, \tag{16}$$

T^* is the characteristic temperature at which correlation effects become important in the nuclear subsystem of the antiferromagnet.[11] Inasmuch as $T_1/T_2 \approx 3$ at the maximum point, this means that on the low-temperature wing of the $T_2^{-1}(T)$ curve correlation effects are important in the dynamics of the nuclear subsystem and consequently important roles are played in T_2^{-1} by both terms [see Eq. (7)]. It must be noted that this seems to follow directly from the fact that the experiment was performed on a sample enriched with ^{57}Fe. The correlation effects should play a lesser role when the active nuclei are diluted: $T^*_{\text{dil}} \propto c_n^{1/2} T^*$. Here c_n is the concentration of the active nuclei.

It follows from (15) and (16) that T_1/T_2 should have a linear dependence on T^{-2}. Figure 3 shows the ratio of the

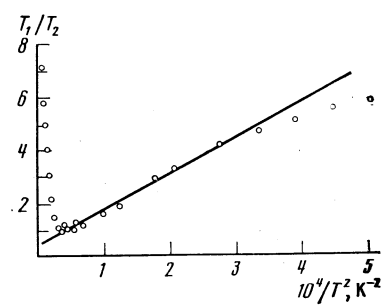

FIG. 3. Dependence of the ratio T_1/T_2 on the square of the temperature.

relaxation times vs T^{-2}. It can be seen that at $T < 250$ K the experimental data can be described by a straight line. Extrapolation of the data to $T \to \infty$ (total neglect of the correlations in the nuclear system) yields $T_1/T_2 = 1/2$, in agreement with the theoretical formula (15). The deviation of the experimental points upward from the line in Fig. 3 at low values of $1/T^2$ ($T^{-2} < 5 \times 10^{-5}$) indicates that at $T > 200$ K (see Fig. 1) the principal nuclear-spin relaxation mechanism is interaction with the magnetic subsystem. The influence of the impurities, which is given by Eqs. (6), (7), and (10), is insignificant in this region. The deviation of the experimental data downward from the line in Fig. 3 in the region $T^{-2} > 5 \times 10^{-4}$ ($T < 45$ K) is due to the fact that the idealized straight line $T_1/T_2 = 1/2 + a/T^2$, $a \propto \varepsilon_0^{-3}$, is valid for the impurity mechanism only under the assumption that the AFMR frequency ε_0 is independent of temperature. The experimental value $a_{\text{exp}} = 1.2 \times 10^4$ deg^2 is of the same order of magnitude as the theoretical estimate $(T^*)^2 \approx 10^4$ K^2. At the same time, the contribution made to the spin-wave gap by the static influence of the paramagnetic impurities (see Eq. (4) of the present paper as well as Refs. 13 and 14) shows that in a sample containing impurities ε_0 actually increases with decreasing temperature. Accordingly, the experimental points of Fig. 3 lie below the idealized line at low T ($T < 45$ K).

At high temperatures, $T > 250$ K, where the impurity relaxation mechanism is inessential, the relaxation rate increases appreciably with rising temperature. Analysis of the experimental data shows that in this region the rates of the spin-spin and spin-lattice relaxations can be represented in the form T_1^{-1}, $T_2^{-1} \approx BT^m$, $m = 5$. This behavior of the relaxation times at high temperatures can be attributed to three-magnon relaxation processes,[15] which yield

$$\frac{1}{T_1^{3m}} = \frac{\eta^6}{S^3} \beta \frac{\omega_n^2}{\omega_E} \left(\frac{T}{\omega_E}\right)^5, \quad \beta \approx 10^{-3}.$$

The difference between the spin-spin and spin-lattice relaxations at $T > 250$ K is due to a manifestation of Suhl-Nakamura broadening in T_2^{-1}:

$$\Delta(1/T_2) = (\Delta\omega_{S-N}^2)^{1/2} \propto c_n^{1/2} \omega_n^2 / \omega_E^{1/2} \varepsilon_0^{1/2}.$$

We note in conclusion that both T_1 and T_2 [see (6) and (9)], which are governed by relaxation absorption in interaction with the subsystem of paramagnetic impurites in antiferromagnets, should depend on the external magnetic field via

the AFMR frequency ε_0. Our experiments were performed at $H = 1$–2 kOe, so that $T_1(H)$ and $T_2(H)$ could not be observed, within the limits of error, because of the appreciable magnetoelastic and impurity gaps in the spin-wave spectrum. It is therefore of interest to study nuclear magnetic relaxation at lower impurity densities in substances with larger Dzyaloshinskiĭ fields (e.g., in $FeBO_3$). Such measurements will permit observation of the field dependence of the relaxation times.

We note also that the difference, by a decade, between T_1 and T_2 in the helium-temperature region indicates that at $T = 4.2$ K the dynamic NMR frequency shift is several times larger than the Suhl-Nakamura linewidth $\Delta\omega_{S\text{-}N}$. Further lowering of the temperature should lead to the presence of nuclear spin waves in the nuclear subsystem in the long-wave region of the spectrum.

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[3)]The interaction of nuclei with paramagnetic impurities will be dealt with in detail separately.

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