# Echo in a system of nuclear spin waves in antiferromagnet $MnCO_3$ with a strong hyperfine interaction

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An investigation was made of the echo in a system of nuclear spins with $k = 0$ and $k \neq 0$ ($k$ is the wave vector) in the antiferromagnet $MnCO_3$. The spectrum of nuclear spin waves formed in this compound because of a strong coupling between the nuclear magnetic and antiferromagnetic systems. The echo was excited by a pair of rf pulses with a carrier frquency equal to twice the spin wave frequency (parametric excitation under parallel pumping conditions). Studies were made of the dependences of the echo amplitude on the external static magnetic field, on the power of the first and second rf pulses, and on the time interval between the pulses in a pair. The echo method was used to determine the rate of relaxation of nuclear spin waves. These relaxation rates were compared with the values deduced from the threshold of parametric excitation of a given type of magnon and with the rate of decay of the free induction signal of parametrically excited nuclear spin waves, which was also determined in the present study. The rate of decay of the free induction signal for $k = 0$ was equal to the value obtained by the other two methods and when $k$ increased, it was faster than the rate of relaxation of spin waves.

## INTRODUCTION

The spin echo methods are used widely in NMR studies.[1] Dumesh and Bun'kov[2] observed the spin echo in systems with a large dynamic shift of the NMR frequency. As shown in Refs. 3 and 4, the mechanisms of formation of the echo in such systems are very different from those in simple nuclear magnetic systems. The dynamic shift on the NMR frequency is determined by the relationship between the nuclear magnetic system and a magnetically ordered electron system. This is manifested most strikingly by antiferromagnets with a weak crystallographic anisotropy and containing $Mn^{2+}$ ions. We investigated the antiferromagnet $MnCO_3$ with the easy-plane anisotropy. The spectrum of low-frequency excitations in this substance, corresponding to the NMR with a dynamic shift and nuclear spin waves, is[5,6]

$$\omega=\omega_n(1-\gamma^2H_\Delta^2/\Omega_{ek}^2)^{1/2}, \qquad (1)$$

where $\omega_n$ is the NMR frequency in a hyperfine field in the absence of the dynamic coupling; $\Omega_{ek}$ is the frequency of antiferromagnetic spin waves; $H_\Delta^2 = 2H_E H_N$ ($H_E$ is the exchange field and $H_N$ is the effective field of the hyperfine interaction created by the nuclear system and experienced by the electron magnetic system). The spectrum of nuclear spin wave given above was checked for the $k = 0$ case in Ref. 7 and confirmed indirectly by a study of parametric excitation of magnons of this type.[8] In our earlier study[9] we observed an echo on excitation with a pair of rf pulses at twice the frequency represented by Eq. (1) and this was done for $k = 0$ and $k \neq 0$. In the present study we obtained detailed information on the behavior of this echo and used it to determine the rate of relaxation of nuclear spin waves in a wide range of wave vectors.

There have been many determinations of the rate of relaxation of spin waves by using the technique of parallel pumping.[10] This technique involves determination of the threshold field of the appearance of parametric excitation of magnon pairs as a function of some external parameter. The magnon lifetime (or rate of relaxation) can be determined using the results of theoretical calculations of the dependence of the threshold field on the parameters of the Hamiltonian, external conditions, and lifetime itself. Although in many cases a good agreement is observed between experimental results and theoretical calculations or models of quasiparticle interactions, there have been frequent objections that this approach is indirect and doubts have been expressed about its results. Since the spin echo method is a direct way of determining the lifetime of magnetic excitations, we shall compare the results of measurements of the rate of relaxation of nuclear spin waves obtained by the echo method with the results deduced using the parallel pumping method. Moreover, we shall compare the two sets of results with the rate of decay of a free induction signal of parametrically excited nuclear spin waves, which was also determined in the present study.

## METHOD

We investigated the echo in a system of nuclear spin waves by applying two rf pulses at a frequency which was double the frequency of excited nuclear spin waves. We called these parametric or Π pulses, because they excited nuclear spin waves parametrically. The duration of the pulses ranged from a fraction of a microsecond to several microseconds, and the time interval between the pulses in a pair was varied from a few to tens of microseconds. The best

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conditions for the parametric excitation of nuclear spin waves were obtained when $\mathbf{h} \| \mathbf{H}$ and $\mathbf{H} \perp C_3$ ($\mathbf{h}$ is a high-frequency magnetic field and $\mathbf{H}$ is an external static magnetic field). In the case of $MnCO_3$ and $C_3$ axis is a third-order crystal symmetry axis of manganese carbonate and it is perpendicular to the easy magnetization axis. A sample of $MnCO_3$ was placed in a helical resonator made of a copper wire 0.3 mm in diameter. The external diameter of the helix was $\sim 4$ mm and its length was $\sim 6$ mm. A resonator with the sample was placed horizontally in a copper tube with an internal diameter 10 mm; the resonator was coaxial with the tube. An external magnetic field created by an electromagnet was directed along the resonator axis. This ensured the orientation necessary for parallel pumping. A sample was oriented using its natural faceting.

The helical resonator with the sample was coupled to two coaxial cables by coupling rods; one of the cables was used to supply microwave power to the resonator and the other to receive the echo signal. A superheterodyne receiver with a preamplifier at its input had a sensitivity of $10^{-13}$ W. The recovery time of the receiver sensitivity after the application of the parametric pulses was 3 $\mu$sec. The use of two master microwave oscillators made it possible to vary independently the amplitudes of the two excitation pulses in a pair. The echo signal detected by the receiver was measured with a sampling voltmeter. This made it possible to determine in the same experiment the behavior of the echo excited by two short $\Pi$ pulses, the free induction signal of nuclear spin waves excited parametrically by a long $\Pi$ pulse, and the threshold field for the parametric excitation of nuclear spin waves either from the appearance of a discontinuity in a pulse or from the appearance of induction after a pulse.

## EXPERIMENTAL RESULTS

Figure 1 shows a record of a video signal obtained from the receiver during echo observations. A gate pulse was used to scan the whole echo observation cycle. The pulses $\Pi_1$ and $\Pi_2$ are the parametric excitation pulses transmitted by the resonator; 1 and 2 are the echo signals. The echo signals were observed in the range of external magnetic fields in which the excitation of nuclear spin waves was possible. In the case of $MnCO_3$ this range was governed by the dispersion law of nuclear spin waves:

$$\omega_{1k} = \omega_p/2 = \omega_n\{1 - \gamma^2 H_\Delta^2 \{\gamma^2[H(H+H_D) + H_\Delta^2] + v^2k^2\}^{-1}\}^{1/2}. \quad (2)$$

The maximum magnetic field corresponded to nuclear spin waves with the wave vector $k = 0$. The following notation is used in Eq. (2): $\omega_p$ is the frequency of the alternating magnetic field, which in the case of manganese carbonate was $\omega_n = 2\pi = 640$ MHz; $\gamma$ is the gyromagnetic ratio: $H_D$ is the Dzyaloshinskii effective interaction field; $v$ is the velocity of antiferromagnetic spin waves in $MnCO_3$. Reduction of the magnetic field from $H_{\text{ref}}$ in the $k = 0$ case increased the wave vector of nuclear spin waves to several units of $10^5$ cm$^{-1}$.

When the wave vector of nuclear spin waves was altered (more precisely, when the external magnetic field was changed in an experiment), the echo signal was affected and it decreased on increase in $k$. Figure 2 shows the dependences of the echo signal power (obtained using a square-law detector) on the external magnetic field obtained for several values of the amplitude of the parametric pulses. On the high magnetic field side the range of existence of the echo signal was bounded by the NMR field at a given temperature and by the frequency $\omega_p/2$. A sharp minimum of the echo amplitude at $H = 0$ was clearly due to demagnetization of the sample. Accoding to the mechanism of excitation of nuclear spin waves,[11] the critical field necessary for the formation of parametric nuclear spin waves should be proportional to $(2H + H_D)^{-1}$, i.e., for $H = 0$ one could expect strong excitation of nuclear spin waves, because $2H$ and $H_0 = 4.4$ kOe were comparable quantities. However, the model describing parametric excitation was developed on the assumption of a single-domain structure in which the sublattice magnetizations are approximately perpendicular to the external static and alternating fields. In fact, in the vicinity of $H = 0$ the sample was demagnetized and there were different directions of the magnetization in the easy plane relative to the polarization of the high-frequency magnetic field.

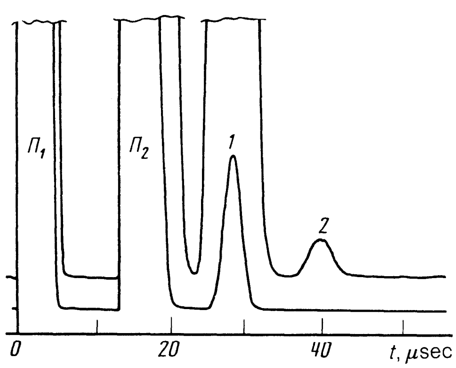


FIG. 1. Characteristic form of the echo signal of a system of nuclear spin waves obtained for $f_p = 1129$ MHz in a field $H = 2.1$ kOe. Here, $\Pi_1$ and $\Pi_2$ are parametric pulses; $\tau_1 = \tau_2 = 2.56$ $\mu$sec; curves 1 and 2 represent the first and second echo signals. The ordinates are shifted relative to one another. The gain in the amplifier for the upper curves was increased by 23 dB. A square-law detector was used.

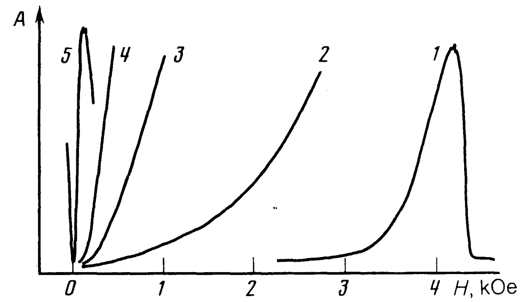


FIG. 2. Dependences of the echo signal amplitude on the external magnetic field applied when the alternating field frequency was $f_p = 1209$ MHz. The amplitudes of the parametric pulses increased from curve 1 to curve 5. The maximum on curve 5 near $H = 0$ corresponded to a shift of the resonator frequency because of magnetization of the $MnCO_3$ sample.

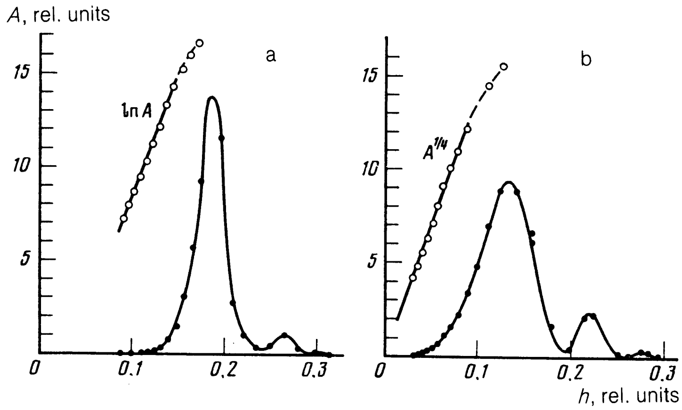


FIG. 3. Dependences of the echo amplitude on the amplitudes of the first (a) and second (b) parametric pulses.

Figure 3a shows the dependence of the amplitude of the echo signal on the amplitude of the first parametric pulse in the case when the amplitude of the second pulse was constant. When the first pulse was applied, parametric excitation of nuclear spin waves began. The number of these waves increased with time in accordance with the law[10]

$$n \propto \exp \{2\eta t(h-h_e)/h_c\}. \quad (3)$$

Therefore, when the amplitude of the first pulse is increased and that of the second is kept constant, the echo signal should rise exponentially, as indeed observed experimentally (Fig. 3). A further increase in the amplitude of the first pulse resulted in saturation of the amplitude of the echo signal, followed by its oscillatory decay. These deviations from the exponential rise of the echo amplitude are due to the onset (and subsequent strengthening) of the phase mechanism that limits the number of spin waves under parallel pumping conditions.[10] This behavior of the echo signal agrees well with the sample-susceptibility oscillations observed on the pulses of the hf power passing through a parallel-pumped resonator containing the sample.[12]

Figure 3b demonstrates the nature of the behavior of the echo signal as a function of the amplitude of the second parametric pulse when the amplitude of the first was constant. It is clear from this figure that during the initial stage there was a rise of the echo signal proportional to $h^4$. A further increase in the amplitude of the second pulse resulted in behavior similar to that observed when the amplitude of the first pulse was varied (Fig. 3a): saturation was followed by decay with oscillations. This occurred when the second pulse interacted strongly with the system of nuclear spin waves and under its influence the number of spin waves becames limited due to the phase mechanism.

## MECHANISM OF ECHO FORMATION

We shall now consider the mechanism of formation of an echo signal due to nuclear spin waves and compare it with the mechanism of formation of the parametric echo considered in Ref. 4. Figure 4 shows the spin configurations which appear in the parametric echo in the former case (upper row) and in the parametric echo due to nuclear spin waves (lower row). In fact, this figure shows the configurations of the combined spin of the two sublattices, but we shall omit the word "combined." At $t = 0$ (Fig. 4a) a resonance pulse at the NMR frequency deflects all the spins by an angle $\alpha$ and by the moment of application of the second pulse the inhomogeneous broadening of the NMR line results in spreading of the spins on a strongly flattened (because of the magnetic anisotropy) cone (Fig. 4b). The second (parametric) pulse applied at $t = t_1$ "deforms" this cone and deflects the spins in the directions of the arrows in Fig. 4b. At $t = 2t_1$ the spins form a pencil (Fig. 4c) and then an induction signal appears at the NMR frequency[4] and at double the frequency.[9]

In the case of parametric excitation of nuclear spin waves the first parametric pulse forms two pencils of spins deflecting them by angles $\alpha$ and $-\alpha$ from the equilibrium position (Fig. 4d). Subsequently, the scenario develops in the same way as in the first case, except that on formation of an echo signal the spins collect into two pencils, as shown in Fig. 4f, and then the echo signal appears at double (relative to nuclear spin waves) frequency, i.e., at the pump frequency.

The spin configurations shown in Fig. 4f may appear also in the parametric echo case considered in Ref. 4, giving rise to additional echo signals which are observed only at the doubled frequency.[9]

Therefore, the mechanism of formation of the echo due to nuclear spin waves is in a sense similar to the mechanism of formation of the parametric echo at the doubled frequency. It is shown in Ref. 4 that the amplitude of the echo signal is proportional to the square of the amplitude of the second parametric pulse and since the signal proportional to the power is recorded experimentally, we find that $P \propto h^4$ (Fig. 3b).

We are now facing the problem related to the spread of the spins after the action of the first pulse. In the case of a system of "free" spins (free in the sense that an hf pulse that deflects them from the equilibrium position is switched off and the spins are left to their own fate), the spreading occurs because of inhomogeneous broadening of the NMR line since the frequencies of the individual spins differ slightly. Otherwise, one would observe a free induction signal which decays with time at a rate $\sim \exp(-t/T_2)$, where $T_2$ is the spin-spin relaxation time. In the case of the parametric exci-

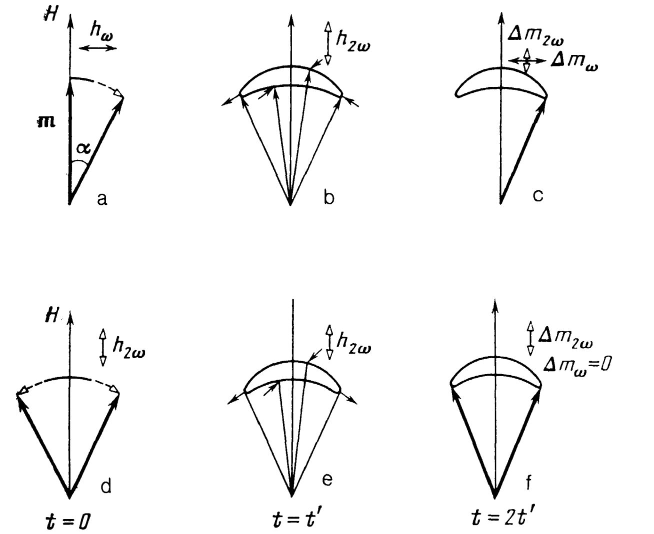


FIG. 4. Spin configurations in the formation of the parametric echo signal (upper row) and of the echo in a system of nuclear spin waves (lower row).

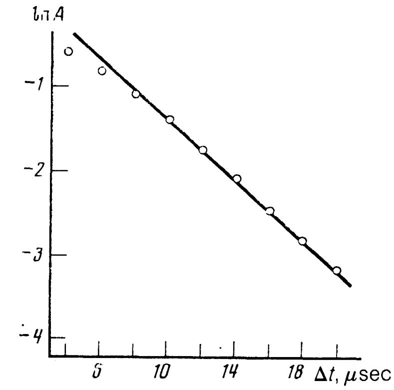


FIG. 5. Dependence of the amplitude of the echo signal on the time between the parametric pulses. A square-law detector was used.

tation, nuclear spin waves with the frequency $\omega_p/2$ are generated (because of the energy conservation law) and the mechanism of excitation is such that these nuclear spin waves are phase-locked to the external alternating field, i.e., the spin waves are excited at a constant frequency (but, in principle, with different wave vectors and the range of such wave vectors is governed by the inhomogeneous broadening of the spectrum of nuclear spin waves). This should give rise to a free induction signal which decays with time and is governed by the magnon lifetime.

However, the excitation of nuclear spin waves in an experiment involves short pulses (of duration amounting to several microseconds), which excite nuclear spin waves in a frequency band $\delta\omega \propto 1/\tau$, where $\tau$ is the duration of a parametric pulse, and this should result in decay of the induction signal with a decay time $\sim\tau$. If nuclear spin waves are excited by an "long" pulse, then the free induction decays at a rate governed by the time $T_2$. It is found that at least for $k = 0$ the decay time of the induction after a long parametric pulse is identical with the decay time of the echo signal, which supports the above conclusions. The problem of behavior of the decay of the free induction signal after a long pulse in the $k \neq 0$ case will be discussed below.

## DETERMINATION OF THE RATE OF RELAXATION OF NUCLEAR SPIN WAVES

The echo due to nuclear spin waves discovered and investigated by us earlier[9] makes it possible to determine the rate of relaxation $\eta$ of nuclear spin waves which is related to the magnon lifetime $T$ by

$$\eta = 1/2\pi T. \tag{4}$$

Figure 5 shows the dependence of the echo signal amplitude on the time interval between the parametric pulses. It is clear from this figure that there is a transition region where the rate of decay of the echo signal varies with time and this region is followed by an exponential decay of the echo signal amplitude. Hence, it follows that immediately after the action of parametric pulses some process takes place in a system of nuclear spin waves and this process distorts the exponential decay law of the number of nuclear spin waves, This process may be, for example, the influence of the free induction field on nuclear spin waves. After the end of a pump pulse these waves decay not in zero external alternating field but in the induction field which decays with time. The intensity of this field may be sufficiently high to have a significant influence on the excited nuclear spin waves, for example, the induction field may be higher than the critical value and this may create new nuclear spin waves. After a certain time when the spins become spread out uniformly over the cone and the induction field becomes sufficiently low, nuclear spin waves begin to decay at a rate governed by the magnon lifetime.

The following points must be borne in mind in determination of the rate of relaxation of nuclear spin waves from data analogous to those presented in Fig. 5. 1) The apparatus records a quantity proportional to the power of the echo signal (square-law detector). 2) The induction field of a sample $h$ is proportional to the number of spin waves $n$ because $f \propto \alpha$, where $f$ is the Bose operator of spin waves, $\alpha$ is the angle of deflection of spins, $h \propto \alpha^2$, and $n \propto |f|^2 \propto \alpha^2$. We therefore find that $P \propto h^2 \propto n^2 \propto |f|^4$, i.e., the echo signal decreases four times faster with time than do the Bose operators of spin waves. In solving the equations of motion for the Bose operators of spin waves[11,13] during the application of a parametric pulse, we find that the following solution is obtained:

$$f \propto \exp\{\eta t(h - h_c)/h_c\},$$

i.e., the rate of relaxation governs the rate of relaxation of the Bose operators, whereas the number of spin waves

$$n \propto |f|^2 \propto \exp\{2\eta t(h - h_c)/h_c\} \tag{6}$$

varies with time at double this rate. 3) The time corresponding to decay of nuclear spin waves excited by the first parametric pulse is equal to the twice the time between parame-

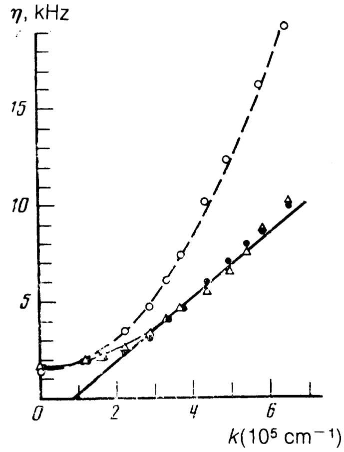


FIG. 6. Dependence of the rate of relaxation $\eta$ of nuclear spin waves on the wave vector $k$ obtained for $f_p = 1177$ MHz at $T = 1.25$ K: Δ) spin echo method: ●) method of parametric excitation of nuclear spin waves; ○) decay of free induction after a long parametric pulse.

tric pulses, i.e., it is equal to the time between the first pulse and the echo signal.

Figure 6 shows the results of measurements of the rate of relaxation of nuclear spin waves obtained by the spin echo method. For comparison, this figure includes also the results obtained by measuring the critical field for the excitation of nuclear spin waves under parallel pumping conditions and also those deduced from a determination of the free induction decay after the end of a long parametric pulse.

The same absolute values are obtained when the rate of decay of nuclear spin waves is determined by the spin echo method and from the decay of the free induction, whereas in the case of parametric excitation of nuclear spin waves calculations require the knowledge of the absolute value of the amplitude of the critical field $h_c$, which cannot be estimated accurately and the error usually amounts to 20–50%. The relative precision is however considerably higher and it is determined by the precision of the attenuator calibration.

As already pointed out above, if $k = 0$, the rate of decay of the free induction signal is identical with the rate of decay of the echo signal. Moreover, if $k = 0$, it is possible to compare these rates of relaxation of nuclear spin waves with the results of measurements of the spin-spin relaxation time $T_2$ obtained by the traditional NMR echo method. Inside a copper screen we placed a single-turn copper wire coil alongside a helical resonator; the coil was used to apply an alternating magnetic field at the NMR frequency to a sample and the coil axis was perpendicular to the resonator axis. Under these conditions the coil and the resonator did not influence one another and the resonator winding did not screen the alternating field at the NMR frequency. Such a coil could be used to determine in a single experiment both the relaxation rate $\eta(k)$ and $T_2$ by the NMR echo method.

It was found that for $k = 0$ the rate of decay of the echo signal due to nuclear spin waves was identical with the rate of decay of the second harmonic of the parametric echo (within the limits of the experimental error). At the NMR frequency the parametric echo signal decayed, as expected ($\Delta m_{2\omega} \propto \Delta m_\omega$), in the same way as the NMR echo,[2] i.e., at half the rate of decay of the echo signal at the doubled frequency.

When the wave vector of the excited spin waves was increased, the free induction signal decayed more rapidly then the echo signal (Fig. 6). Clearly, there was some mechanism which was activated on increase in the wave vector $k$ of the excited spin waves and which increased the rate of free induction decay. One should point out that the observation of the free induction signal after a long parametric pulse made it possible to detect the moment of onset of the parametric excitation of nuclear spin waves and consequently provided a highly sensitive method for the determination of the critical field $h_c$.

Since the critical field amplitude $h_c$ was subject to a considerable systematic error, an attempt was made to compare the results obtained on parametric excitation of nuclear spin waves with the results of measurements of the magnon lifetime by the spin echo method using renormalization of the critical field amplitude. It is clear from Fig. 6 that after renormalization of $h_c$, a fairly good agreement was obtained between the dependences $\eta(k)$ determined by the two methods. Hence, at least in the case of nuclear spin waves the rate of relaxation could be found from the data on the parametric excitation of these quasiparticles.

We shall now consider the difference between the rate of decay of an echo in a system of nuclear spin waves and the rate of decay of the induction after parametric excitation by a long pulse. We shall assume that the free induction decay is governed by the change in the number of the excited spin waves and, additionally, by some spin-wave-pair dephasing that depends on the wave vector. We shall postulate that the mechanisms of these two processes are independent, i.e., that the total rate of decay of the induction is the sum of two decay processes, which makes it possible to determine the difference between the rates of decay of the induction and the nuclear spin-wave echo. Figure 7 shows the dependence of the square root of this difference on the wave vector of the excited spin waves. It is clear from this figure that the additional rate of relaxation of the induction associated with dephasing is proportional to the square of the wave vector:

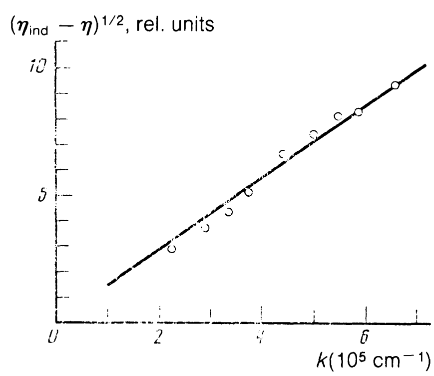


FIG. 7. Dependence of $(\eta_{\text{ind}} - \eta)^{1/2}$ on the wave vector $k$.

$$(\eta_{ind} - \eta) \propto k^2, \quad (7)$$

where $\eta_{ind}$ is the rate of decay of the free induction signal.

Dephasing of parametrically excited spin waves has been observed earlier for the antiferromagnets $MnCO_3$ and $CsMnF_3$ in the case of parametric excitation of electron spin waves.[14]

## CONCLUSIONS AND FINAL COMMENTS

We investigated the spin echo in a system of parametrically excited pairs of nuclear spin waves. The data on the echo signal decay yielded the rate of relaxation of nuclear magnons with different wave vectors. It was found that variation, within a reasonable scale, of the dependence of the rate of relaxation on the wave vector obtained from the results of parametric excitation of nuclear spin waves under parallel pumping conditions described well the echo method results. The rate of relaxation could then be described by the expression

$$\eta = (0.012 \pm 0.002) kT \ [\text{Hz}] \quad (8)$$

Since the echo measurements made it possible to determine the rate of relaxation of nuclear spin waves directly from the experiments, the main error in the above expression is our nominal value of the wave vector. This is primarily due to inhomogeneous broadening of the NMR line, which makes the magnetic field corresponding to a homogeneous resonance (i.e., to $k = 0$) indeterminate, and also due to the error indicated actually in Eq. (8).

An investigation of free induction at the doubled nuclear spin wave frequency (i.e., at the pump frequency) after the action of a long parametric pulse revealed that the decay of the signal was faster then the echo decay. This was attributed to a mechanism causing dephasing of parametrically excited magnon pairs under the action of dipole fields. The dephasing rate dependende on the wave vector as follows:

$$\eta_{ph} = 2.8 \cdot 10^{-8} k^2 \ [\text{Hz}]. \quad (9)$$

These measurements were carried out at a fixed temperature ($T = 1.25$ K).

Recording of the free induction signal provided a highly sensitive method for the determination of the critical field in parametric excitation of nuclear spin waves.

The questions why the rate of relaxation of nuclear spin waves was half that predicted theoretically by Richards,[15] and which dephasing mechanism applied in the case of free induction, remain unanswered.


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