Magnetoacoustic excitation of nuclear spin echo

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The results of experiments on the excitation of nuclear spin echo on I^{127} nuclei in CsI by two- and three-pulse combinations of electromagnetic and acoustic pulses are discussed. The limitation of the vector model in the interpretation of the excitation of echo signals is demonstrated. The experimental results can be explained by the theory proposed in the present paper, in which a multiquantum nature of echo formation is assumed.

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1. INTRODUCTION

Observation of nuclear spin echo is carried out at the present time by a wide variety of methods of investigation of rapidly occurring processes in matter. The use of resonant acoustic pulses could give additional information, in comparison with electromagnetic excitation, on the dynamics of the transition processes in spin systems. This is especially important for conducting media and superconductors. However, the acoustic excitation of the spin echo has been realized so far only for the electron spins of Fe^{2+}, Fe^{3+}, Mn^{2+} and Ni^{2+} in MgO, as a consequence of great experimental difficulties. [1]

In the present work, we discuss the results of the first observation of nuclear spin echo that is excited by a combination of electromagnetic and acoustic pulses. [2]

The theoretical consideration of the excitation of spin inductions and echo by resonant acoustic pulses began with the researches of Koloskova, Kopvillem and Kessel'. [3] Two differences in principle from the case of electromagnetic excitation were observed [14]; 1) the acoustic pulses interact preferentially with the electric quadrupole moments of the nuclei; 2) the sound wave length is usually smaller than the dimensions of the investigated sample.

The first feature must be taken into account in the determination of the power supplied by the acoustic pulses that is necessary for the effective reversal of the magnetic moment of the nuclei. The acoustic pulse does not interact directly with the spins, but "reverses" the quadrupole moment of the nuclei, which is strongly coupled with the magnetic moment.

The second feature is the basic reason for the nonsymmetrical theoretical conclusions on the possibility of observation of electromagnetic nuclear spin echo, excited by the acoustic pulses. The experimentally observed quantity in this case is the transverse component of the magnetization of the sample, which represents the algebraic sum of the transverse components of the magnetic moments of the nuclei. It is evident that, during the time of action of the acoustic pulse, the nuclear magnetic moment takes a phase connected with the phase of the acoustic wave at the given point. The conclusion can then be drawn that the contribution to the total magnetic moment from a layer of sample of thickness equal to the acoustic wavelength λ is equal to zero. It turns out that the contribution to the signal of the nuclear spin echo can be made only by a small layer of the sample, of thickness less than $\lambda/2$ (maximum signal from $\lambda/2$). Such a conclusion is supported by a calculation which uses kinetic equations of the Bloch type. [4]

However, it was shown there that the acoustic pulses can strongly affect the longitudinal component of the magnetization. The experimental study of this component after the action of the pulse can give information on the transition processes.

It is known that pulsed excitation of transition processes in nuclear spin systems, creating signals that are sufficiently large to be amenable to detection, is possible only upon satisfaction of a number of conditions:

1. The duration of the exciting pulses (Δt_i) and the interval between pulses (τ_i) should be much shorter than the time of longitudinal relaxation (T_1).

2. The acoustic pulses with duration Δt should essentially reverse the magnetic moment of the nuclei (up to π), i.e., the condition

$$2\pi\frac{3}{16I(I+1)}G\varepsilon\Delta t\sim 1$$

should hold (see Ref. 4). In this equation, I is the spin of the nucleus, G is the constant of dynamic spin-phonon coupling, ε is the amplitude of the acoustic deformations.

Starting from this, we selected the spin system of the I^{127} nuclei ($I=5/2$) in single crystals of CsI ($G=1600$ MHz). The spin system was excited by pulses of a variable magnetic field (M pulses), by resonant transitions $\Delta M=\pm 1$ in the spectrum of the system (M is the magnetic quantum number), and by acoustic pulses (A), resonant transitions $\Delta M=\pm 2$. At certain combinations of M and A pulses, echo signals were observed in addition to the effect of the A pulses on the magnetic induction.

In contrast to the experiments of Ref. 1, where both electromagnetic and acoustic echo signals were observed, only electromagnetic echo signals were recorded in the present research. This is associated with

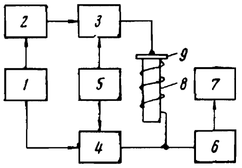

FIG. 1. Block diagram of the spectrometer. 1—master oscillator, 2—frequency doubler, 3—Transmitter of A pulses, 4—transmitter of M pulses, 5—pulse sequence former, 6—receiver, 7—oscilloscope, 8—sample, 9—quartz piezotransducer.

the smallness of the deformations of the nuclear acoustic echo signals in comparison with the electronic, as a consequence of the significantly smaller nuclear constants of spin-phonon coupling.

The following experimental facts were the most unexpected, not corresponding to semiclassical representations of the character of echo signal formation.

1. An anomalously large value of the ratio of the intensities (I) of the echo signals in MA and MM sequences $I_{MA}/I_{MM} \sim 1$ (in the scale for $\tau \to 0$). The theory predicted a value of $I_{MA}/I_{MM} \sim \lambda/2l$, [4] equal to about 10^{-2} for our conditions.

2. Independence of the intensity and phase of the spin echo signal, excited by the MA sequence, of the phase of the second pulse.

The stated features of the observed signals of the ordinary and stimulated echoes are explained by drawing on the proposition of the multiquantum character of the excitation of spin systems in the superconducting state, in which the echo signals can be generated.

2. EXPERIMENTAL TECHNIQUE

The measurements were carried out on a pulse spectrometer, the block diagram of which is shown in Fig. 1. For the excitation of the magnetic and acoustic pulses at frequencies of 10 and 20 MHz, respectively, we used two transmitters 3 and 4 with power up to 50 W, which could operate in coherent and noncoherent modes. In the coherent mode, a master oscillator 1 (G4-44) was used with the frequency doubler 2. In the noncoherent mode, two separate master oscillators were used. The pulse sequence shaper 5 allowed us to obtain any combination of A and M pulses in two and three-pulse sequences. The pulse lengths could be changed from one to 100 microseconds.

As a source of the ultrasound, we used the quartz transducer 9 with a resonant frequency of 20 MHz. To make the acoustic contact of the transducer with the sample 8, we used silicone oil GKZh-94. The amplitude of the high-frequency voltage at the transducer amounted to 50 V, while the estimate of the amplitude of the acoustic deformations by the method of equivalent electrical impedance[5] gave a value of $\varepsilon \sim 2\times 10^{-4}$.

A sample of cylindrical shape, diameter 7 mm, length 30 mm, was placed in a constant magnetic field perpendicular to the axis of the cylinder, which coincided with the [111] direction of the crystal lattice. The time of passage of the A pulse through the sample was 50 μsec. For excitation of M pulses and reception of the signals of induction and echo, we used a single-coil pickup, in which the sample was placed.

The detector 6 operated at a frequency of 10 MHz and allowed us to receive a signal of free induction from the I^{127} nuclei in CsI with a signal/noise ratio of 1/3. All the measurements were carried out at room temperature.

To verify the acoustic nature of the interaction of the spin system with the A pulses, we carried out control experiments:

1. It is seen in Fig. 2a that the A pulse in the MA sequence not only forms an echo signal, but also materially changes the value of the free induction signal from the M pulse. When the acoustic contact between the transducer and the sample is broken by introduction of a paper liner, not only is an echo not formed, but there are no noticeable traces of interaction of the second pulse with the signal of free induction (Fig. 2b).

2. The insignificant reaction of the detector to the action of the A pulse shows the absence, at the moment of application of the A pulse, of an electromagnetic field with frequency 10 MHz, corresponding to the transition $\Delta M = \pm 1$.

3. THEORY AND INTERPRETATION

Experimental and theoretical studies of the excitation of nuclear spin echo signals in systems with spin $I > \frac{1}{2}$ by electromagnetic pulses, resonant to various transitions in the energy spectrum, have been produced in a number of researches. [6,7] An important difference in the calculation of the interaction of the spin system with the acoustic field from the interaction with the electromagnetic field consists in the difference in the wavelengths. Therefore, attention is concentrated on those features which bring into consideration the smallness of the acoustic wavelength in comparison with the dimensions of the sample.

Calculations, by the standard method of the evolution operator, [8] carried out by us for specific conditions

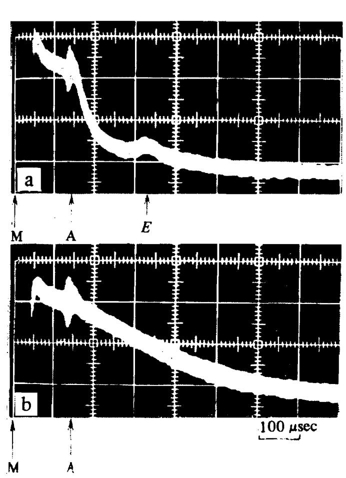

FIG. 2. Nuclear induction and echo signals in MA sequence of pulses: a) in acoustic contact between the piezotranducer and sample, b) in disruption of the acoustic contact.

(I^{127} in CsI) show the possibility of observation of the spin echo signals at just this moment of time and with those characteristics which were obtained experimentally. Therefore, it is necessary to find out in what is the exact nature of the unexpectedness of the obtained experimental results.

In the consideration of the nuclear spin echo, for nuclei with spin $\frac{1}{2}$, the vector model, which describes the interaction of spins with the external magnetic field, is standard. According to this model, the phase of the transverse component of the spin is determined by the phase of the external field. A similar model is implicitly assumed also in the consideration of pulse excitation of many-level systems, including excitation by sound. In the latter case, the phase of the spin should depend on the phase of the acoustic wave at the given point. But such a representation is in contradiction with the experimentally observed independence of the intensity and phase of the spin echo signals, excited by the MA sequence, of the phase of the acoustic pulse. These features, and also the value of the echo, can be explained by assuming that all the spins interact with the A pulse identically, independent of the phase of the acoustic wave at the given point of the sample.

For clarification of the reason for the non-correspondence of the model with reality, we trace the change in the phase characteristics of the transverse component of the magnetization of the spin system (spin $I > \frac{1}{2}$) in its excitation by the acoustic pulse. We can write the Hamiltonian of the interaction of the spin j with the acoustic wave of frequency ω, with resonance transition $\Delta M = \pm 2$ (M is the magnetic quantum number) in the form

$$\mathscr{H}_A{}^j = a(\hat{I}_+{}^j)^2 \exp\{-i(\mathbf{k}\mathbf{r}_j - \omega t + \varphi_A)\} + a^*(\hat{I}_-{}^j)^2 \exp\{i(\mathbf{k}\mathbf{r}_j - \omega t + \varphi_A)\}, \tag{1}$$

where $|a|$ is the constant of interaction of the spin with the acoustic field, $\mathbf{r}_j$ is the radius vector of the j-th spin, $\mathbf{k}$ is the sound wave vector, φ_A is the initial phase of the acoustic pulse. For I^{127} ($I = 5/2$), the gyromagnetic ratio is positive; consequently, the operators $(\hat{I}_\pm^j)^2 \exp\{\mp i(\mathbf{k}\cdot\mathbf{r}_j - \omega t + \varphi_A)\}$ describe the transitions $\Delta M = \pm 2$ in the energy spectrum of the j-th spin with emission (absorption) of a phonon of frequency ω with wave vector $\mathbf{k}$ and phase $(\mathbf{k}\cdot\mathbf{r}_j + \varphi_A - \omega t)$. For the parts of these operators which describe the phase characteristics of the phonons, we introduce the notation

$$b_{\mathbf{k}\varphi}{}^j = \exp\{i(\mathbf{k}\mathbf{r}_j + \varphi_A)\} = \exp\{i\psi_k{}^j\},$$
$$(b_{\mathbf{k}\varphi}{}^j)^+ = \exp\{-i(\mathbf{k}\mathbf{r}_j + \varphi_A)\} = \exp\{-i\psi_k{}^j\}. \tag{2}$$

Let the initial state of the spin be characterized by the density matrices $\rho^j_{\alpha\beta}(0)(\alpha, \beta = -5/2, -3/2, -1/2, 1/2, 3/2, 5/2)$. The state of the spin after the action of the acoustic pulse of duration Δt (Δt is shorter of the times of reversible and irreversible processes) is determined by the density matrices

$$\hat{\rho}^j(\Delta t) = (\hat{\mathscr{L}}^j(\Delta t))^{-1}\hat{\rho}^j(0)\hat{\mathscr{L}}^j(\Delta t),$$
$$\hat{\mathscr{L}}^j(\Delta t) = \exp\{i\hbar^{-1}\Delta t \hat{\mathscr{H}}^j_{A\,\mathrm{int}}\}, \tag{3}$$

where $\mathscr{L}^j(\Delta t)$ is the evolution operator, and consideration is usually carried out in the interaction representation. In this case the time dependence simply disappears in the Hamiltonian $\hat{\mathscr{H}}^j_{A\,\mathrm{int}}$. According to the theory of matrix functions, [9] the matrix $\hat{\mathscr{L}}(\Delta t)$ can be represented in the form of a linear combination of successive powers of the matrices $\hat{\mathscr{H}}^j_{A\,\mathrm{int}}$ from zero to $(l-1)$ (l is the order of the matrix $\hat{\mathscr{H}}^j_{A\,\mathrm{int}}$; in our case it is equal to 6).

Since $\hat{\mathscr{H}}^j_{A\,\mathrm{int}}$ has only $(\alpha, \alpha \pm 2)$ non-zero elements, we can show by direct calculation that there will be $(\alpha, \alpha + 2n)$ nonzero elements of the matrix $\hat{\mathscr{L}}(\Delta t)$, where $n = -2, -1, 0, 1, 2$. Introducing in these matrix elements the terms which depend on the phase characteristics of the phonons, we can obtain

$$\mathscr{L}^{\pm}_{\alpha,\alpha+2n} = \mathscr{A}^{\pm}_{\alpha,\alpha+2n}(b_{\mathbf{k}\varphi}{}^j)^n, \tag{4}$$

where

$$(b_{\mathbf{k}\varphi}{}^j)^{-n} = (b_{\mathbf{k}\varphi}{}^{j+})^n, \quad (b_{\mathbf{k}\varphi}{}^j)^0 = 1.$$

The transverse components of the magnetization of the spin system are experimentally observable quantities, proportional to the expressions

$$\mathrm{Sp}\sum_j \hat{\rho}^j(t) I^j_{x(y)}, \tag{5}$$

where $\hat{\rho}^j(t)$ is the density matrix of the j-th spin at the instant of time of interest to us. Summation is carried out over all spins. Since the matrices $\hat{I}^j_{x(y)}$ have only $(\alpha, \alpha \pm 1)$ nonzero matrix elements, only the elements $\rho_{\alpha,\alpha\pm1}(t)$ of the matrix density make a contribution in Eq. (5). Using (3), (4), we write out the expressions for the matrix elements of the density matrix in which we are interested, after the effect of the acoustic pulse:

$$\rho^j_{\alpha,\alpha+1}(\Delta t) = \sum_{n,m} \mathscr{A}^{-1}_{\alpha,\alpha+2n}\rho^j_{\alpha+2n,\alpha+1-2m}(0)\mathscr{A}_{\alpha+1-2m,\alpha+1}(b_{\mathbf{k}\varphi}{}^j)^{n+m}. \tag{6}$$

Analyzing the values of the transverse components of the magnetization of the individual spins and of the entire sample with the aid of Eq. (6), after action of the acoustic pulse, we can draw the following conclusions.

1) The transverse magnetization does not arise if, before the action of the A pulse, the spins are in the state described by the diagonal density matrix. Actually, only the nondiagonal elements of the spin density matrix before the action of the pulse enter into the right side of Eq. (6).

2) The transverse magnetization of the individual spin represents the sum of five components with phases 0, $\pm\psi^j_k$, $\pm 2\psi^j_k$, where 0 corresponds to the value of the phase of the transverse spin magnetization under the action of the A pulse. The values of the phases are determined by the factors $(b^j_{k\varphi})^{n+m}$ in the expression (6) and their number is the possible set of numbers $(n+m)$.

3) The transverse magnetization of the sample is determined on the basis of the algebraic sum of the components of the magnetization of the sums of "phase 0," since the four other components make a small contribution in the summation over the sample (see the Introduction). The appearance of such a component of the

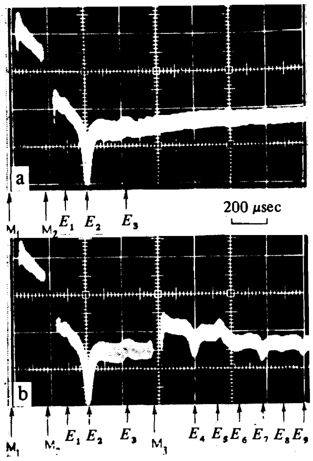

FIG. 3. Echo signals in the excitation of a spin system by electromagnetic pulses: a) MM sequence, b) MMM sequence.

magnetization again means that each spin interacted with the A pulse equally, independent of the phase of the acoustic wave at the given point.

A physical interpretation can be given to the possibility of the existence of such an interaction by assuming that each matrix element of the evolution matrix operator (4) characterizes one of the possible processes of transition between the spin energy levels with absorption or the radiation of phonons in the time of the A pulse. The number n again characterizes the number of phonons taking part in the process (an "n-quantum" process). In such an interpretation, the appearance of a component of "phase 0" is due to the process in which the spin absorbs and radiates an identical number of phonons ($n = -m$ in Eq. (6)). In a two-level system of such type of process, only the diagonal elements of the density matrix can change, i.e., the longitudinal component of the magnetization. In a multilevel system, the nondiagonal elements of the density matrix also changes, i.e., the transverse component of the magnetization, since the transitions can take place between different pairs of levels.

It must be emphasized that, by requiring here a representation of the multi-quantum nature of the process of interaction of the spin system with the A pulse, we have used only the phase characteristics of the phonon field, but have assumed the energy of the acoustic wave to be unchanged. Such a semiclassical description is possible because of the high power of the acoustic pulses and their comparatively weak interaction with the spin system.

Since only one of the five components of the magnetization of the spins takes part in the formation of the transverse component of the magnetization of the spin system after the A pulse, only that one can take part in the formation of the echo.

The work of Alekseev *et al.* must be noted, [10] in which the times of generation of the echo signals and the wave vectors of each signal echo were determined in the case of the acoustic excitation of a spin system with different values of the spin. Special attention is drawn to the result contained in the research, namely, that the appearance of an echo signal with a wave vector independent of the wave vector of one of the exciting pulses is possible. However, the discussions of the reasons for the appearance of such a result and the possibility of its experimental realization are not given in the paper.

4. RESULTS

On the basis of the theory set forth above, we have analyzed the spin echo signals excited by sequences of two and three pulses. In the excitation in any sequence of several echo signals, the amplitude of each of them is characterized by their dependence on the duration of the exciting pulses. Experimentally, the durations of pulses were selected in which the maximum number of echo signals were observed for each sequence. The pulse durations given below correspond to precisely such conditions. We make use of the notation: Δt_i is the duration of the ith pulse, τ_i the distance between the i-th and $(i+1)$-st pulses, $s(t)$ is the width of the echo signal at the instant of time t.

1. MM sequence of the exciting pulses; $\Delta t_1 = 5.5\mu$ sec, $\Delta t_2 = 3.3$ μsec, $\tau_1 = 225$ μsec. Three echo signals (E) were observed (Fig. 3a) at the instants of time $t(E_1) = (3/2)\tau_1$, $t(E_2) = 2\tau_1$, $t(E_3) = 3\tau_1$. $s(E_1) = 20$ μsec, $s(E_2) = s(E_3) = 40$ μsec. The decay of the free induction signal with time of 400 μsec is shown by the presence in the spin system of two times of reversible relaxations. In correspondence with the theoretical analysis of Refs. 6, 7, the decay of the induction is due to the dipole mechanism of reversible relaxation, and the formation of echo signals that we have observed is due to a quadrupole mechanism. Here the signal E_2 is formed by the simultaneous action of both relaxation mechanisms. Therefore, the decay of the amplitudes E_1 and E_2 with increase in τ_1 is characterized by the time of dipole reversible relaxation ($T_d \sim 300$ μsec), and the decay of the amplitude of E_2 is determined by the time of the irreversible transverse relaxation of the spin system, which is equal to $T_2 \approx 2.5$ μsec in this experiment. It is known[7] that other echo signals should form in the spin system considered, due both to dipole and quadrupole mechanisms of reversible relaxations. We have not observed them because of the insufficient sensitivity of the apparatus.

2. MMM sequence: $\Delta t_1 = 5.5$ μsec, $\Delta t_2 = 3.3$ μsec, $\Delta t_3 = 4$ μsec, $\tau_1 = 225$ μsec, $\tau_2 = 815$ μsec. Of greatest interest in the three-pulse sequence is the stimulated echo, [11] which allows us to determine the longitudinal relaxation time T_1. A series of echo signals (Fig. 3b) is observed, among which were two stimulated at the instants of time $t(E_4) = \tau_2 + 2\tau_1$ and $t(E_6) = \tau_2 + 3\tau_1$. The relaxation time $T_1 \approx 15$ μsec is determined in the change of τ_2.

3. MA sequence; $\Delta t_1 = 7$ μsec, $\Delta t_2 = 22$ μsec, $\tau_1 = 120$ μsec. An echo signal is observed (Fig. 4a) at the time $t(E_1) = 2\tau_1$ with width $s(E_1) = 40$ μsec, coinciding with the signal widths of E_2, E_3 in the MM sequence. The echo intensity does not change if the phase of the second pulse is changed arbitrarily. In correspondence with the interpretations set forth in the previous paragraph, the

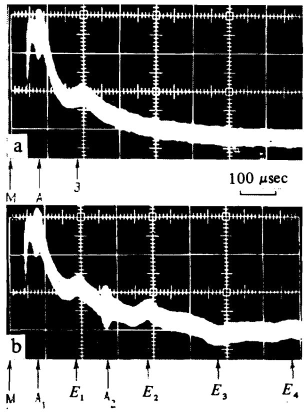

FIG. 4. Echo signals: a) in MA sequence, b) in MAA sequence.

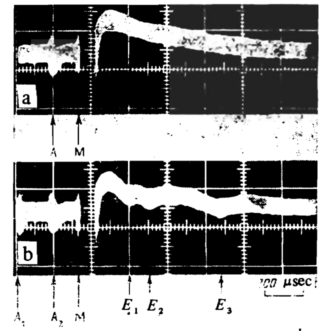

FIG. 5. Stimulated echo signals: a) AM sequence, b) AAM sequence.

formation of the echo is due to the existence of magnetization of "phase 0" after the action of the A pulse. Direct calculation of the time of formation of the echo signal due to this component shows that the formation of the echoes brought about by the quadrupole mechanism of reversible relaxation.

4. MAA sequence: $\Delta t_1 = 7.0$ sec, $\Delta t_2 = \Delta t_3 = 15$ μsec, $\tau_1 = 120$ μsec, $\tau_2 = 240$ μsec. Echo signals are observed (Fig. 4b) at the times $t(E_1) = 2\tau_1$, $t(E_2) = 2\tau_2$, $t(E_3) = 3\tau_2$, $t(E_4) = 4\tau_2$, with width $s(E_1) = s(E_2) = 40$ μsec, $s(E_3) = s(E_4) = 60$ μsec.

As is seen from Fig. 4b, the phase of the signal E_3 differs from the phase of the remaining signals. The theoretical calculation shows that no stimulated echo is excited in the given sequence. The observed signals are simply additional echo signals.[11] The different phases of the echo signals are explained, as also in the MMM sequence, by the multilevel nature of the system. It is interesting that the location of the echo signals after the third pulse does not depend on τ_1.

5. AAM sequence; $\Delta t_1 = \Delta t_2 = 15$ μsec, $\Delta t_3 = 1.9$ μsec, $\tau_1 = 120$ μsec, $\tau_2 = 60$ μsec. Echo signals are observed (Fig. 5b) at the times: $t(E_1) = 2\tau_1 + \tau_2$, $t(E_2) = (5/2)\tau_1 + \tau_2$, $t(E_3) = 4\tau_1 + \tau_2$, $s(E_1) = s(E_2) = 40$ μsec, $s(E_3) = 80$ μsec. Calculation shows that this single sequence of three pulses with participation of the acoustic, in which formation of a signal of stimulated electromagnetic echo is possible at the instant $(3/2)\tau_1 + \tau_2$, $2\tau_1 + \tau_2$, $(5/2)\tau_1 + \tau_2$, $3\tau_1 + \tau_2$, $4\tau_1 + \tau_2$. At the moment $3\tau_1 + \tau_2$, the stimulated echo should be formed by the simultaneous action of both mechanisms of reversible relaxation. The experiment on the observation of the echo in the change of τ_2 confirms the result that all the observed signals are stimulated echoes.

6. In AA, AAA, and AM sequences (Fig. 5a), the echo signals are not recorded. Calculation shows that the electromagnetic echo should not be formed.

7. In the sequences MAM, MMA, AMM and AMA, the observed echo signals do not differ in principle from those discussed above.

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