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NMR inversion echo analog in cobalt and lithium ferrite under the combined action of radiofrequency and magnetic pulses

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A comparative study of the NMR inversion echo analog (magnetic echo) formation in the rotating coordinate system in multidomain samples of cobalt and lithium ferrite has been carried out under the action of a magnetic videopulse applied between two radio-frequency pulses exciting the two-pulse echo and in the case of the combined action of the magnetic videopulse and radio-frequency pulse, leading to the formation of a magnetic echo. The magnetic echo signal appearance is related to the domain wall displacement when the magnetic videopulse amplitude exceeds the domain wall pinning force. A correlation is shown between the results of determining the pinning force and domain walls mobility by these two methods, which allows one to use these alternative methods for measuring the domain wall pinning force and mobility in magnets.

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Keywords: NMR, inversion echo, magnetic videopulse, domain wall, pinning, lithium ferrite, cobalt

1. Introduction

Nuclear magnetic resonance (NMR) in ferromagnets, enhanced by hyperfine interaction, was discovered in 1959 [1]. Currently, NMR is one of the most powerful methods for studying the structure and properties of magnetically ordered substances [2–4].

A remarkable feature of the manifestation of NMR in magnets is that in many cases the main contribution to the intensity of resonance absorption is made by nuclei located in domain walls (DWs). Since the DW is easy to control when exposed to magnetic videopulses (MVP), their use is a convenient technique for studying the features of the formation of additional echo signals arising under the influence of MVP [5].

The most important factor that should be taken into account in the NMR of magnetic materials is the electron-nuclear hyperfine interaction (HFI) resulting in a huge local field (LF) H_n on a nuclear site [2]. Due to it in these systems, the theory and the methods of NMR are very different from other materials. For a nuclear spin system, the HFI is equivalent to the action of a local magnetic field $H_n = A \cdot M$ on the nuclear magnetization, where M is the electron magnetization, A is the HFI factor. The LF value in magnets reaches $\sim 10^5 - 10^6$ Oe. Thus, the resonant frequency of NMR in magnets is mainly determined by H_n, in contrast to NMR in non-magnetic materials. In addition, the external radio-frequency (RF) field with the amplitude of H_1 and the frequency close to NMR induces the forced oscillations of electron magnetization M. Thanks to the LF, this creates the oscillating fields of enhanced amplitude ηH_1 on nuclei, where η is RF field amplification factor of the order of $10^2 \sim 10^3$ in magnetic domains, but much greater amplification occurs in the DWs ($\sim 10^5$) of magnets and this is a signal from nuclei in DWs, which is usually studied. Thus, the applied RF field as well as the observed signals increase in η times. For this reason, for studying NMR in magnets the NMR spectrometers are in some respects simpler than conventional NMR spectrometers, but they should be able to operate in a wide frequency range, because NMR line widths in multidomain magnets are usually of the order of tens of MHz, and in some cases from tens to hundreds of MHz, at the average carrier frequency in the range of $30 - 1000\,\mathrm{MHz}$.

Under the action of the applied MVP, the DWs are rapidly accelerated to the speed of several hundred m/s. There is a trend towards finding the ways to further increase these speeds in order to increase the speed of memory and logic technologies.

For the first time, the dynamics of DW in a single-crystal ferrite sample grown in the form of frame under the MVP action was studied by Galt [6]. The motion of the DW was induced under the influence of the MVP supplied to the primary coil wound on one side of frame. The induction signal arising from the motion of the DW was recorded by the second coil wound on the opposite frame side and observed on the oscilloscope screen. It was shown that the dynamics of the DW is described by the linear dependence of the DW velocity υ on the applied MVP amplitude H:

$$\upsilon = S(H - H_0), \tag{1}$$

where S is the mobility of the DW, and H_0 is the pinning field below which the DW is fixed.

Since the external RF field acts on the nuclei through the electronic subsystem, the explanation of the considered phenomena should be based on the idea of what kind of motion the electronic magnetic moments M undergo in the DWs under the action of the MVP. Even an insignificant displacement of DW can be accompanied by a large rotation of M: in cobalt, at the MVP amplitude of $\sim 200\,\mathrm{Oe}$ and the duration $\tau_d \sim 0.5\,\mu\mathrm{s}$, the DW shift can be of the order of its thickness, and then some moments inside the 180° DW will complete the rotation. In this case, the rotation angle of $\mathbf{M}$ inside DW is proportional to the displacement of DW. This process is accompanied by the change in LF on nuclei proportional to the DW displacement, due to the anisotropy of LF in cobalt [7].

In [8, 9], using the excitation of the nuclear spin system in thin ferromagnetic films of polycrystalline cobalt under the sequential action of RF and MVP pulses, the so-called inversion mechanism of echo signal formation was realized. It was associated with a change in the direction of the precession of nuclear spins at a change in the orientation of the electron magnetization. In the initial state, the sample was magnetized along the anisotropy axis and the nuclear spin system was in equilibrium.

The RF pulse deflects the nuclear magnetization from the equilibrium position, then the nuclear isochromates are dephased due to the inhomogeneities of of LFs. When exposed to MVP at the time of $t = \tau$, the sample was remagnetized over the time of $\tau_R \sim 1\,\mathrm{ns}$. After the magnetization reversal, the direction of the LF changes to the opposite one, which also leads to the change in the direction of isochromate precession and, accordingly, to their rephasing at the time of 2τ with the formation of the inversion echo signal. The condition for the formation of an inversion echo is the fulfillment of the relation [10]

$$\omega_j \tau_R \ll 1, \tag{2}$$

were w_j is the angular velocity of the precession of the j-th isochromate, this is equivalent to the fulfilment of the condition $\tau_d \ll T_p$, where T_p is the precession period of nuclei. As the NMR frequency in a polycrystalline cobalt film $\nu_{\mathrm{NMR}} = 218\,\mathrm{MHz}$ at $T = 77\,\mathrm{K}$, this condition is fulfilled at $\tau_R \sim 1\,\mathrm{ns}$ and $T_p \sim 5\,\mathrm{ns}$, which is observed experimentally (during the magnetization reversal time τ_R, the nuclear magnetization practically does not change own orientation).

As it is shown in [10], in addition to the possibility of exciting the inversion echo signal upon the direct rotation of $\mathbf{M}$ in a magnetized thin magnetic film, implemented in [8], the possibility of obtaining it from nuclei located in DWs at a rapid rotation of $\mathbf{M}$ as a result of the fast DWs displacement under the action of MVP was indicated. As shown in [10], the generation of an additional echo signal under the action of MVP pulse is technically much easier to realize in the case of nonadiabatically fast change of the direction of effective magnetic field in the rotating coordinate system (RSC) at the combined action of RF and MVP, since in this case the following relation should be fulfilled:

$$\Delta\omega_j' \cdot \tau_d \ll 1, \tag{3}$$

where $\Delta\omega_j' = \sqrt{\Delta\omega_j^2 + \omega_1^2}$ is the angular velocity of the precession of the j-th isochromate in RCS relative to the effective magnetic field $\mathbf{H}_{\text{eff}} = (\Delta\omega_j \cdot \mathbf{z} + \omega_1 \cdot \mathbf{y})/\gamma_n$, as in Fig. 1 shown. Here γ_n is the nuclear gyromagnetic ratio, $\mathbf{z}$ and $\mathbf{y}$ are the unit vectors in the RCS, $\Delta\omega_j = \omega_j - \omega_{\text{rf}}$ is the detuning for the j-th isochromate, $\omega_1 = \gamma_n \eta H_1$ is the amplified RF magnetic field in frequency units, H_1 is the RF pulse amplitude, η is the gain of RF field, which reaches $\sim 10^3$ in cobalt and $\sim 10^5$ in lithium ferrite, τ_d is the MVP duration.

Note, that the condition (3) can be fulfilled at much longer $\tau_d \sim 0.1\,\mu$s, since under the nonresonant excitation usually $\Delta\omega_j' \ll \omega_j$. Echo signal of this type called as magnetic echo (ME), was observed in cobalt micropowder [11].

The important role of RF pulse edges and of MVP action in the formation of additional echo signal at the combined action of RF and MVP pulses [10] is due to the fact that just at these time moments the fast changes in the direction of $\mathbf{H}_{\text{eff}}$ in the RCS take place.

This can be understood in frames of nonresonant mechanism of single pulse echo (SPE) formation and its extension to the model of formation of multipulse excitation analogs in frames of SPE method [10,12]. Multipulse excitation analogs in the frames of SPE method in multidomain magnets are observed upon sudden jumps of $\mathbf{H}_{\text{eff}}$ in RCS occurring at the action of RF pulse, including as well the MVP action.

The nonresonant mechanism of SPE formation was first proposed in [13], and further was extended to the case of formation of multipulse excitation analogs in SPE method [10, 12]. According to the nonresonant model of SPE formation, the stepwise (nonadiabatic) switching on of the RF field results in the appearance of an angle between the equilibrium direction of nuclear magnetization and the direction of effective field $\mathbf{H}_{\text{eff}}$ in the RCS and, then, in the precession of magnetization (isochromates) around $\mathbf{H}_{\text{eff}}$ in the RCS. An abrupt RF field switching off changes of the direction of $\mathbf{H}_{\text{eff}}$, causing the isochromates to reverse phases in the RCS and the form of the SPE. In case of the formation of multipulse excitation analogs of SPE the RF pulse edges and MVP, where the fast changes of $\mathbf{H}_{\text{eff}}$ directions take place, are equivalent to the RF pulses in the method of multipulse excitation of Hahn spin echo [10, 12].

The aim of this work is to carry out a comparative study of the analogs of inversion echo signals in the RCS arising under the combined action of RF and MVP pulses on nuclear spin system in the DWs of polycrystalline samples of cobalt and lithium ferrite.

2. Experimental results and their discussion

The measurements were carried out on a phase-incoherent spin echo spectrometer in the frequency range of 40–400 MHz at the temperature of 77 K. A standard self-excited generator was used in the frequency range of 40–220 MHz. The oscillator frequency can be smoothly tuned using various inductors and tuning capacitors. A two-wire line commercial Lecher-type generator

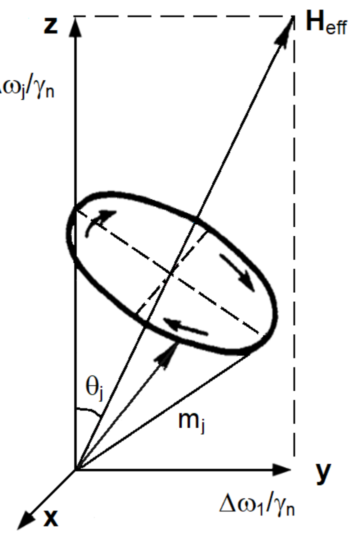

Figure 1. Precession of nuclear magnetization vector $\mathbf{m}_j$ of j-th isochromate around the effective field $\mathbf{H}_{\text{eff}}$ (1) in a rotating coordinate frame, $\Delta\omega_j > 0$.

including two inductors with different number of turns was used in 200–400 MHz range. For the pulse lengths in the range from 0.1 to 50 μs, the maximum amplitude of the RF field obtained on the sample was about 3.0 Oe, and the steepness of the fronts was no worse than 0.15 μs. The dead time of receiver was $\sim 1\,\mu$s.

The scheme of the experiment on pulsed action is shown in Fig. 2. The pulsed magnetic field was created by a gated current stabilizer of adjustable amplitude and by an additional copper coil, making it possible to obtain the magnetic field pulses of the order up to 500 Oe at the sample size of $\sim$ 10 mm. The samples of lithium-zinc ferrite $Li_{0.5}Fe_{1.0}Zn_{0.15}O_4$, were studied, having the form of rings with the diameter of 12–15 mm and the mass of 5.8 g, enriched in the ^{57}Fe isotope to 96.8% for increasing the intensity of the echo signal. The polycrystalline cobalt powders, obtained by fusion in the inductive furnace, were also used having the average grain size less than 50 μm [13].

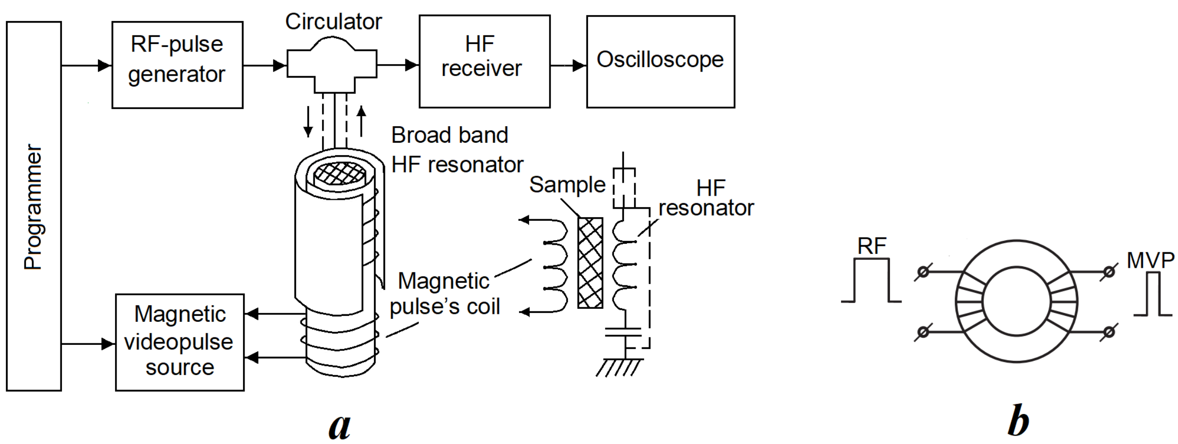

Figure 2. (a) Schematic of the experiment; (b) NMR cell in the case of lithium ferrite.

Let us investigate the effect of MVP on the intensity of a two-pulse echo (TPE) when it is exposed to MVP action between two RF pulses or between the second RF pulse and the echo signal, Fig. 3a (so-called asymmetric action) [14]. The NMR spectrum of investigated face-centered cubic (FCC) cobalt micropowder is presented in Fig. 3b. At relatively small amplitudes of MVP, its effect is reduced to a reversible displacement of the DWs. In magnets with anisotropic LF, the NMR frequency depends on the position of the nucleus within the DW. When the DW is displaced under the action of MVP, the positions of the nuclei within the DWs and, accordingly, their NMR frequencies are changed. This leads to a partial violation of the phase coherence of the precessing nuclear spins and reduces the efficiency of their rephasing process leading to the decrease of echo intensity. It is natural to associate the MVP amplitude at which the echo begins to decrease with the onset of DW displacement, Fig. 4.

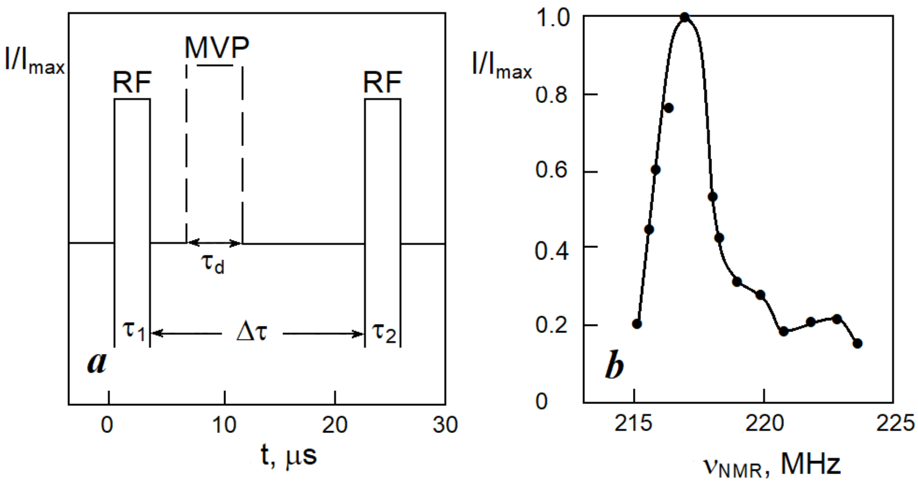

Figure 3. (a) Location of the MVP relative to the RF pulses; (b) NMR spectrum of FCC cobalt at $T = 77\,\mathrm{K}$.

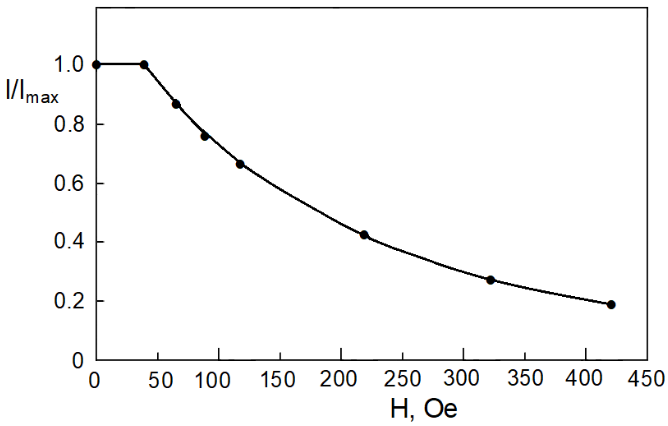

Figure 4. Dependences of the echo intensities on the amplitude of the magnetic videopulse in the case of cobalt micropowder, $\nu_{\mathrm{NMR}} = 217\,\mathrm{MHz}$, $\tau_1 = \tau_2 = 1\,\mu\mathrm{s}$, $\tau_d = 0.5\,\mu\mathrm{s}$, $T = 77\,\mathrm{K}$.

The anisotropy of LF is especially pronounced in Co, which has a much higher LF anisotropy as compared to lithium ferrite [7, 16]. Therefore, the effect of MVP on TPE in cobalt is most effective at the asymmetric action of MVP, in contrast to lithium ferrite having by an order of magnitude lower value of LF anisotropy [15, 16]. But instead of this lithium ferrite is characterized by much higher values of its DW mobility as compared with cobalt. As a result of which η factor in lithium ferrite is about $\eta \sim 2 \cdot 10^5$ as compared to only $\eta \sim 100$ in cobalt [15].

Let us now present results of the excitation of inversion echo analog in the RCS (ME) in cobalt micropowder (see Fig. 5).

The additional ME signal arises, Fig. 5b, as a result of a rapid nonadiabatic change of the direction of $\mathbf{H}_{\text{eff}}$ when the position of the nuclei in the DW is changed with its displacement by the MVP and, consequently, by the associated changes in the LF and enhancement factor η, when the MVP amplitude exceeds a certain threshold value H_0 associated with a pinning force, see line 2 in Fig. 5c. The SPE decreases correspondingly, see line 1 in Fig. 5c, showing the redistribution of contributions of excited nuclei between two types of echoes. This ME signal could be considered as an analog of inversion echo in RCS [11]. Line 2 in Fig. 5c shows the dependence of intensity of the stimulated ME, formed by the MVP and the two edges of the RF pulse in analogy with the three-pulse stimulated Hahn echo, on the MVP amplitude.

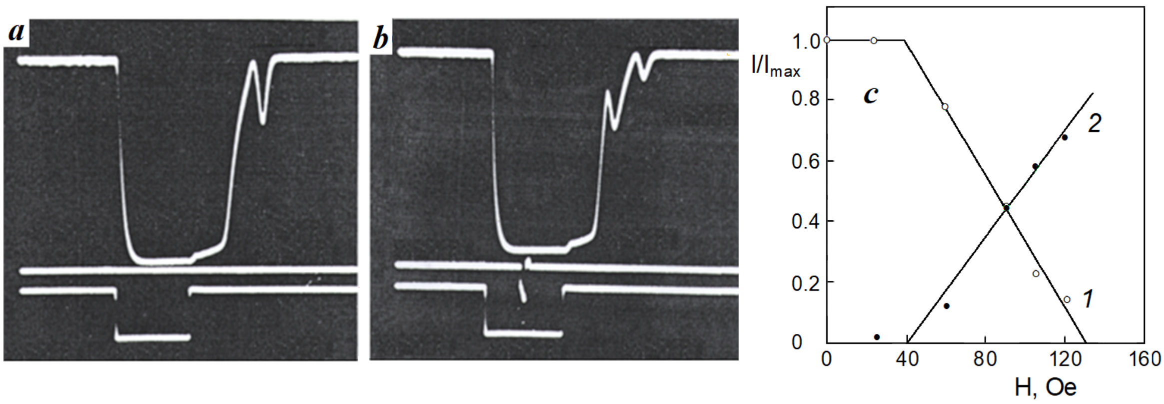

Figure 5. (a) Oscillogram of a single-pulse echo (SPE) signal following the induction signal decay on the upper beam in Co; (b) oscillogram of a magnetic echo (ME) signal in Co, formed by a magnetic videopulse and two edges of the RF pulse, followed by the SPE signal. The middle beam shows the location and duration of the magnetic videopulse. The lower beam represents the RF pulse: $\nu_{\text{NMR}} = 216\,\text{MHz}$, $\tau = 18\,\mu\text{s}$, $T = 77\,\text{K}$; (c) dependences of the echo signal intensities in Co on the amplitude of the magnetic videopulse, $\nu_{\text{NMR}} = 216\,\text{MHz}$, $\tau = 22\,\mu\text{s}$, $\tau_d = 0.5\,\mu\text{s}$, $T = 77\,\text{K}$: line 1 – SPE; line 2 – stimulated magnetic echo (ME) signal. Figs (b) and (c) are taken from our work [11].

The comparison of Figs 5c and 4 clearly shows for the first time the close values of pinning forces measured by these methods and confirms that the mechanism of the formation of ME signal is really connected with the displacement of DWs under the action of MVP.

The establishment of the form of the corresponding diagrams of the effect of MVP in lithium ferrite in connection with much lower values of its LF anisotropy and higher mobility of DWs is of the great interest. The NMR spectrum of lithium ferrite at $T = 77\,\text{K}$ consists of two well-resolved lines, where the low-frequency line belongs to the tetrahedral A sites, and the high-frequency line to the octahedral B sites Fig. 6a. The dependences of TPE signals on the MVP amplitude applied between RF pulses is shown for $\nu_{\text{NMR}} = 71\,\text{MHz}$ and $\nu_{\text{NMR}} = 74\,\text{MHz}$ frequencies related to A and B positions, correspondingly. It is also observed a significant increase of the mobility of DWs up to the one order of magnitude, and the decrease of pinning force in lithium ferrite as compared to cobalt. A particularly large effect of MVP on TPE signal Fig. 6b, is observed for the echo signal from nuclei located in octahedral B positions of lithium ferrite at the frequency of 74 MHz with more anisotropy of LF [16], as compared to the echo signal from nuclei in tetrahedral A positions at the frequency of 71 MHz. The analysis of the dependences

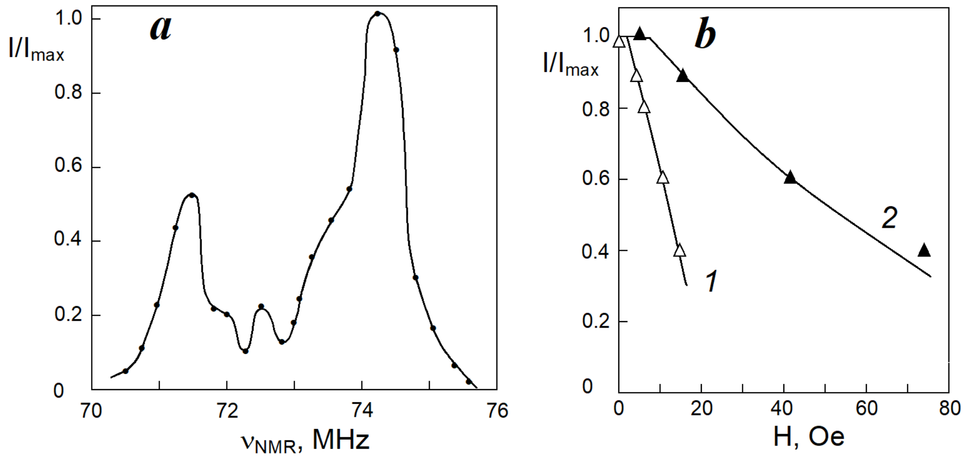

Figure 6. (a) ^{57}Fe NMR signal spectrum in lithium-zinc ferrite; (b) Two-pulse echo (TPE) signal dependences at MVP asymmetric action on nuclei arranged in DWs in B (1) and A (2) positions. Curve 1 – $\nu_{\rm NMR} = 74$ MHz, Curve 2 – $\nu_{\rm NMR} = 71$ MHz, $\tau_1 = \tau_2 = 1\,\mu$s, $\tau_d = 0.5\,\mu$s, $T = 77$ K.

of the effect of combined action of RF and MVP pulses on echo signals in the studied samples shows the possibility to generate ME signals in lithium ferrite similar to those in cobalt when the MVP amplitude exceeds the value of pinning force leading to fast displacements of DWs and hence to the stepwise changes of $\mathbf{H}_{\rm eff}$ in RCS.

The oscillogram of observed stimulated ME signal in lithium ferrite, formed after the induction signal decay and accompanied by SPE, is presented in Fig. 7a, and the studied dependencies of their intensities on MVP amplitude are shown in Fig. 7b being similar to those in cobalt Fig. 5c.

The MVP amplitude at which the ME signal appears, Fig. 7b, correlates with the MVP amplitude acting on the TPE, at which there begins their decrease. The reduction is related with the DW pinning force Fig. 6b, which gives an alternative way of measuring the DW pinning force H_0 and the DW mobility in magnets.

The observed experimental dependences of ME, SPE and TPE signals can be understood taking into account that, according to (1), under the action of the MVP, the DWs reversibly shift at the distance of Δx proportional to the amplitude of MVP $\Delta x = \upsilon \cdot \tau_d = S(H - H_0)\tau_d$, when the MVP amplitude H exceeds the value of pinning force H_0. Under the combined action of RF and MVP. the nuclei in Δx layer experience the effect of an abrupt change of the magnitude and the direction of the effective magnetic field $\mathbf{H}_{\rm eff}$ in RCS due to the changes of LF and η. Hence, accordingly to the nonresonant mechanism of SPE formation, the action of MVP is equivalent to the effect of a second RF pulse at the formation of stimulated Hahn echo, leading to the formation of stimulated echo-response called as ME [11].

In this case, its amplitude is proportional to the number of nuclei in the Δx layer, formed at the displacement of DW: $I_{ME} \sim \Delta x/L$, where L is the width of a DW section excited by RF pulse. Correspondingly, these nuclei do not contribute to SPE, reducing it to $I_{\rm SPE} \sim (L - \Delta x)/L$. In this case, the jump-like change of the nuclear NMR frequencies in RCS must satisfy the condition $\Delta\omega_j'\tau \ll 1$ (3), so the period of precession of nuclei $T_{\rm eff}$ in RCS should be much larger as compared to τ_d being fulfilled at $\tau_d \sim 0.5\,\mu$s under our experimental conditions. At the action of MVP on the TPE within the interval between RF pulses, the RF field is absent and nuclei precess in LFs at frequencies $\omega_j = \gamma_n H_{\rm LF}$, In this case the condition $\omega_j\tau_d \ll 1$ should be fulfilled. This condition (similar to (2)) should be performed requiring the nanosecond duration of MVP

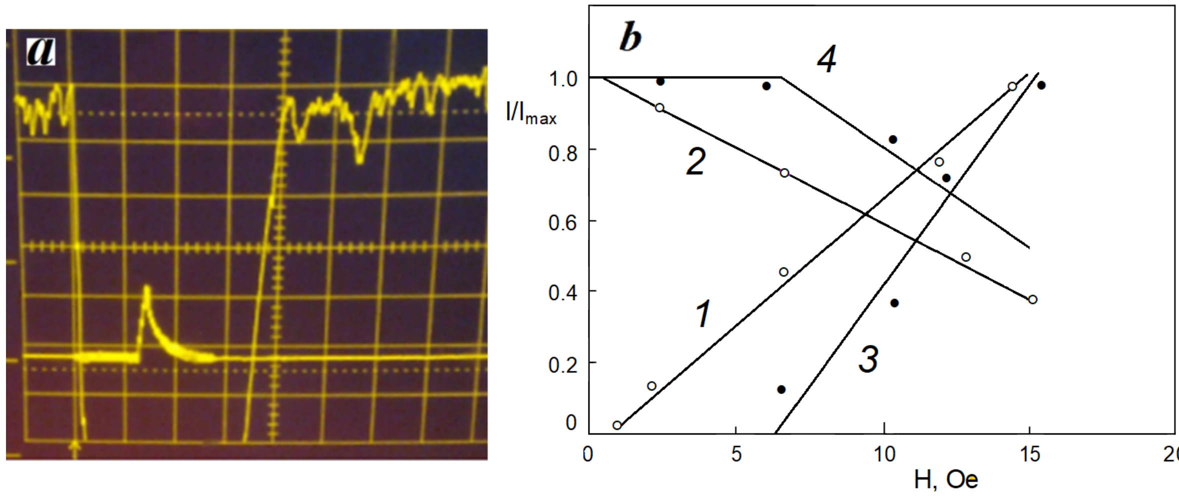

Figure 7. (a) Oscillogram of magnetic echo (ME) signal, formed after the induction signal decay, and followed by the single-pulse echo (SPE) signal in lithium-zinc ferrite (upper beam), $\nu_{\mathrm{NMR}} = 71\,\mathrm{MHz}$, $T = 77\,\mathrm{K}$, the lower beam shows the duration of the RF magnetic pulses, as well as the amplitude of the magnetic videopulse; (b) dependences of the magnetic (lines 1 and 3) and single-pulse (lines 2 and 4) echoes on the amplitude of the magnetic videopulse (MVP) at frequencies 74 and 71 MHz, correspondingly; $\tau_{\mathrm{RF}} = 15\,\mu\mathrm{s}$, $\tau_d = 0.5\,\mu\mathrm{s}$, $T = 77\,\mathrm{K}$.

as in the case of inversion echo in order to have additional ME signals [10]. Therefore, in our experimental conditions the effect of MVP on TPE leads only to a decrease in the intensity I_{TPE} of TPE. The reduction due to the loss of phase coherence of the nuclei located in the Δx layer is proportional to the displacement of the DW $I_{\mathrm{TPE}} \sim (L - \Delta x)/L$. This qualitative consideration allows one to understand the obtained experimental dependences of the ME, SPE and TPE signals under the influence of MVP.

In the above described experiment by Galt [6] for a single-crystal sample of ferrite $\mathrm{Ni}_{0.75}\mathrm{Fe}_{0.25}\mathrm{O}_4$, a value close to 0.1 Oe for H_0 was obtained. Earlier, in the classical Sixtus-Tonks experiment [17] the value H_0 of about 5 Oe was obtained for magnetization reversal of a homogeneous wire of 380 micron diameter made of $\mathrm{Ni}_{14}\mathrm{Fe}_{86}$ alloy under the action of MVP and with an inductive recording of the DW passage.

In the magneto-optical analogue of the Sixtus-Tonks method [18] the value of H_0 close to 22 Oe was measured for the permalloy $\mathrm{Ni}_{80}\mathrm{Fe}_{20}$ nanowire of 200 nm wide and 5 nm thick.

Thus, our estimates of the value of H_0 in lithium ferrite fall within the range of values obtained by the above mentioned methods, and the average value of H_0 in lithium ferrite is approximately one order of magnitude less than that obtained by us for cobalt. The greater value of the pinning force in the case of cobalt can be understood taking into account the fact that the value of the RF field gain factor η in cobalt, which is proportional to the magnetic susceptibility related to the DW displacements is by 2-3 orders of magnitude less than that of lithium ferrite [2].

3. Conclusion

The ME signals (inversion echo analogs in RCS) were excited in DWs of polycrystalline multidomain samples of cobalt and lithium ferrite at the combined action of RF and MVP pulses. Their appearance is related with the onset of DW motion when MVP amplitude exceeds the DW pinning force. The comparative study of pinning and mobility of DWs in cobalt and lithium ferrite has been carried out under the action of an MVP applied between two RF pulses on the TPE, and in the case of combined action of RF and MVP pulses, leading to the formation of

ME signals. A correlation is shown between the results of determining the pinning force and DW mobility using these two alternative methods for measuring the DW pinning force and the mobility in these magnets. The estimations of DW pinning force made by these NMR methods are compared with some results obtained by other methods and can be used for similar measurements in other magnetic materials, in particular, in structures with the scale of the order of micrometer and submicrometer for applications in recording media and magnetic random access memory (MRAM).

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