Study of domain wall pinning in the rare earth-free permanent magnets using nuclear magnetic resonance method

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Abstract

The possibility of using of the modified nuclear magnetic resonance (NMR) technique for studying of the domain wall pinning in cobalt micro powders and micro- and nanowires is demonstrated. In particular, information about the domain wall pinning in cobalt nanowires is useful for the fine tuning of the magnetic properties of the rare earth-free magnets.

The pinning force of domain walls was studied by NMR two-pulse echo method in combination with an additional magnetic video-pulse depending on the magnetic video-pulse duration and an outer steady magnetic field value in cobalt micro powders for the first time. The comparative study of the two-pulse echo dependence on the magnetic video-pulse amplitude in cobalt micro- and nanowires was also carried out.

Keywords: Nuclear spin echo, magnetic video-pulse, two-pulse echo, rare earth-free permanent magnets, domain wall mobility.

Introduction

In order to qualitatively improve the environmental characteristics of modern aircraft (noise level, toxicity of exhaust gases from internal combustion engines), which is related to the gradual implementation of strict environmental norms established by IATA (International Air Transport Association) and ICAO (International Civil Aviation Organization) of the world aviation industry developed the concept of "all-electric aircraft".

An "all-electric aircraft" is an aircraft that does not have internal combustion engines and all of its propulsion equipment is powered by electricity. To create thrust in such aircraft, electric motors are used, which are powered by on-board electrical energy sources (batteries, supercapacitors, fuel cells, solar cells, etc.) and transmit mechanical power to propellers (fans).

Fig. 1 Electric airplane Extra 330LE with Siemens electric engine

The feasibility of creating an "all-electric aircraft" is due to:

with the development of power electronics and the development of a powerful semiconductor converter device and non-contact commutation and protection equipment based on them.

By processing new magnetic materials and creating powerful and compact electric motors and electric generators based on them, their characteristics exceed existing analogs.

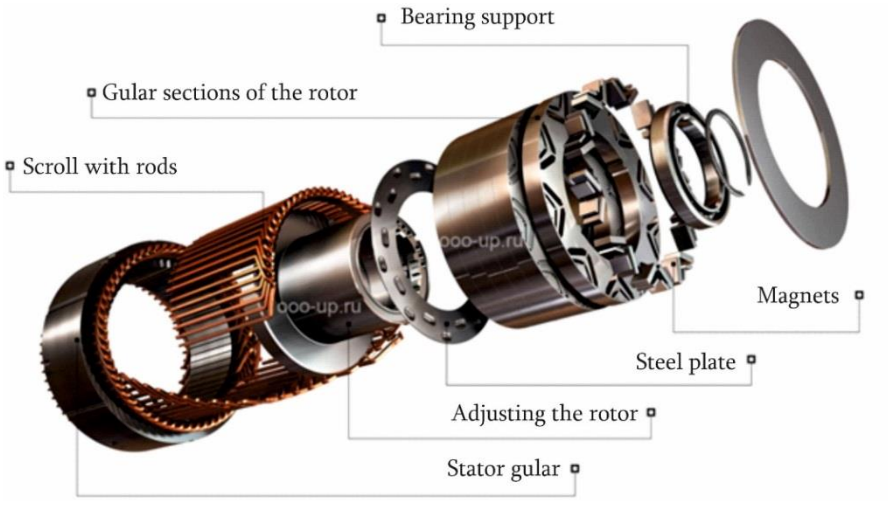

Fig. 2 Synchronous electric motor with built-in permanent magnets

Currently, the field of application of permanent magnets in technology is very wide because they represent an autonomous source of a permanent magnetic field (without the supply of electrical energy), which significantly improves the mass-size and operational characteristics of the electric motor-generator device.

There are natural and artificial permanent magnets. Natural permanent magnets are obtained from the mineral magnetite. Artificial permanent magnets are used to improve magnetic characteristics (magnetic induction B, coercive force - H_{cb}, maximum energy product - BH_{max}, maximum operating temperature - T_{max}, Curie point - Tcur).

Artificial permanent magnets are made from various metals (cobalt, iron, nickel, etc.) and alloys containing rare earth metals (samarium-cobalt; neodymium-iron-boron) and they are magnetized. Magnets of this type maintain their magnetized state for a long time.

The negative side of these permanent magnets is their stiffness and sensitivity to temperature. As a result of strong heating, they completely lose their magnetic properties.

Currently, the country producing permanent magnets containing rare earth elements is mainly China.

Due to the recent significant increase in the share of electric aircraft in aviation, the demand for permanent magnets will increase further, and their cost will increase accordingly. Therefore, theoretical-experimental studies of samples of permanent magnets containing rare earth metals developed with new technologies are very important.

Magnets containing rare earth metals can be produced using different technologies (chemical, precipitation method, shock wave hot deposition method, etc.)

In this scientific work, such an actual issue related to the study of domain wall pinning of permanent magnets not containing rare earth metals is discussed using the nuclear magnetic resonance method.

Nuclear magnetic resonance (NMR) in magnets is currently a powerful microscopic method for characterizing various magnetic materials [1-4]. The spontaneous magnetization of a ferromagnet such as cobalt polarizes s-electrons, creating an effective hyperfine field (HFF) on nuclei through the Fermi contact interaction (a brief discussion of the origin of the effective field and other features of the NMR methods in magnets is given in [1]). The existence of an effective field on nuclei makes it possible to observe NMR in the absence of an external constant field, which is necessary when observing NMR in non-magnetic materials. The HFF reaches a value of the order of 10^5 - 10^6 Oe. The typical NMR linewidth in nonmagnetic materials is about 1 Oe, while in ferromagnetic materials the linewidths range from about 10^2 Oe in iron to greater than 10^3 Oe for dilute impurities in iron. The detection of NMR in magnets is greatly facilitated by the fact that the RF field acts on the nuclei through electronic magnetization. This leads to an RF field amplification by the enhancement factor $\eta_d \sim 10^2$ within the domains and to a much stronger amplification effect in a DWs with $\eta_w \sim 10^4$. For this reason, magnetic NMR spectrometers are in some respects simpler than conventional NMR spectrometers. However, they must be tuned over a wide frequency range up to 1 GHz due to the broad NMR lines in magnets. A remarkable feature of the manifestation of NMR in magnets is that in many cases the main contribution to the intensity of resonant absorption is made by nuclei located in the DWs. Since DWs are easy to control under the action of magnetic video-pulses, their use is a convenient method for studying the features of the formation of additional echo signals arising under the action of a magnetic video-pulse (MVP) [4].

One of its advantages is the ability to provide valuable information about the properties of domain walls (DWs). The inclusion of additional magnetic video-pulses (MVPs) capable of causing a displacement of DWs makes it possible to study the pinning force (degree of pinning) and the mobility of the DWs, in particular, in cobalt nanowires used for fabrication of rare earth-free magnets [5], providing information useful for a fine tuning of their magnetic properties [6,7].

For the first time, the dynamics of DWs under the action of MVP in a single crystal of a ferrite sample grown in the form of a frame was studied by Galt [8]. It was shown that the DW dynamics is described by a linear dependence of the DW velocity υ on the amplitude of the applied MW pulse H:

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

where S is the mobility of the DW, H_0 is the pinning force (the critical field below which the DW is fixed).

In [6], the spin echo NMR technique in combination with MVP acting on a lithium–zinc ferrite sample was used to study the characteristics of DW pinning centers in lithium–zinc ferrite. In particular, in [6], the effect of a long MVP overlapping the rephasing and reading RF pulses of a two pulse echo (TPE) and stimulated echo (SE) signals, respectively, was studied, as well as the interval of their rephasing between these RF pulses and echo signals.

The suppression of echo signals occurs due to the partial loss of phase coherence of isochromats due to the change in their local fields caused by the displacement of DWs and the RF field amplification factor η under the action of MVP [9].

In [6] (Fig. 3) the dependence of the normalized TPE amplitude on the amplitude of the pulsed magnetic field is given for two values of the external magnetic field: $H_e= 0$ and $H_e = 1000$ Oe, and in [7] (Fig. 1 b) it is given its modified dependence showing that the results of [6, 7] also indicate the possibility of measuring the DW pinning force H_0 by the NMR method, which is determined by the MVP amplitude, below which the DWs are pinned.

This assumption was tested in [7] using two alternative methods for measuring the DW pinning strength: the first of them uses the action of a short MVP ~ 1 μs long, acting in the interval between a pair of RF pulses, on a TPE signal [9]. In this case, H_0 was determined by the value of the MVP, after which the TPE signal began to decrease due to the displacement of the DWs caused by the exposure to MVP. In the second case. H_0 was determined from the amplitude of the MVP, acting from a combination with an RF pulse, leading to the formation of a signal of the so-called magnetic echo signal (ME) [10].

In this work, the methodology of [6,7,10] is used for the study of the dependence of the pinning force H_0 in cobalt on the duration of MVP τ_m and the magnitude of the external magnetic field H_e. As known [11], cobalt and lithium-zinc ferrite are very different in their NMR properties: in cobalt, the anisotropy of the HFF is an order of magnitude higher than its value for lithium ferrite. In addition, the value of the NMR amplification factor η in lithium ferrite is about 10^3 times higher than the value of amplification factor η in cobalt, which indicates a much greater mobility of DW in lithium-zinc ferrite as compared to cobalt.

We can obtain a preliminary estimate of the dependence of H_0 on the duration of MVP from Fig. 1, obtained on the basis of Fig. 2 from [9], if it is modified similarly to Fig. 1 from [7], taking into account the presence of DW pinning force H_0.

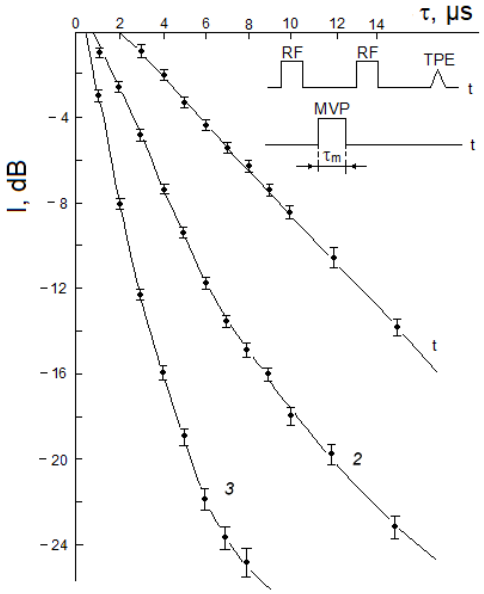

Fig. 3 Dependence of TPE (A –sites of nickel ferrite) on the duration of MVP τ_m at H = 2 Oe (1), 4 Oe (2), 10 Oe (3).

Fig. 1 shows the dependence of TPE (for A-sites of nickel ferrite) on the duration of MVP at H = 2(*1*), 4(*2*), 10 Oe (*3*). Analysis of the dependence of H_0 on τ_m in this figure shows that for τ_m and H_0 (representing the intersection points of dependences *1*, *2* and *3* with the axis τ_m in Fig. 1), the relation $A_m = H_0 \cdot \tau_m$=const takes place, for all H_0, i.e. the pinning force H_0 is inversely proportional to τ_m. The value A_m, which is the area of the MVP, is constant for all threshold values of τ_m, when the TPE intensity begins to decrease due to the tear-off from the pinning centers.

We also note that earlier in [6,7,10] the dependence of H_0 on the external magnetic field H_e was not studied.

As known [6], the application of a large constant magnetic field reduces the number of DWs, and the remaining ones are distributed over pinning centers contributing to greater pinning.

The purpose of this work is to study the experimental dependence of H_0 on τ_m and H_e on the example of the effect of MVP on the TPE signal observed from ^{59}Co nuclei in the DWs of cobalt micropowder and compare them with similar results obtained for cobalt micro- and nanowires.

Experimental results and discussion

The measurements were carried out on a phase-incoherent spin echo spectrometer [12] in the frequency range of 200–400 MHz at a temperature of 293 K. In the range of 200–400 MHz, a commercial Lecher-type generator with a two-wire line, including two inductors with different numbers of turns, was used. For pulse lengths in the range from 0.1 to 50 μs, the maximum amplitude of the RF field produced on the sample was about 3.0 Oe, and the front steepness was no worse than 0.15 μs. Receiver dead time ~ 1 μs.

The scheme of the experiment on pulsed magnetic action is given in [7, 12]. The MVP was created by a gated current stabilizer of adjustable amplitude and an additional copper coil, which made it possible to obtain magnetic field pulses of the order of 500 Oe for a sample size of ~10 mm.

Cobalt micropowders were obtained by the alloying method [13] with an average grain size of ~10 μ. Cobalt microwire samples were synthesized under the influence of an external magnetic field of 500 Oe using the electroless chemical deposition technique [5].

Commercial cobalt nanowire samples from Plasma Chem GmbH with an average diameter of 200–300 nm and a length of up to 200 μ were also studied by this method.

Characteristic parameters of RF pulses: duration - a few microseconds, a delay between them - tens of microseconds, a carrier frequency of 213 MHz at T = 293 K coincides with the frequency of the nuclei in the center of the DW of the face-centered cubic (fcc) phase of cobalt.

The scheme of the experiment is shown in Fig. 2.

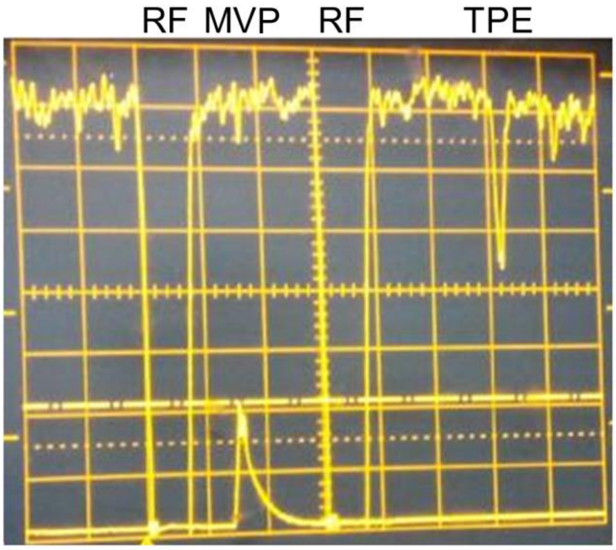

Fig. 4. Oscillogram of the TPE signal in cobalt (upper beam), the lower beam is a wave-meter signal showing the position of the RF and MVP pulse

Let us present the results of the study of the pinning force H_0 under the action of an additional MVP, depending on the duration of the MVP τ_m, on the TPE signals in cobalt micropowder, Fig. 3.

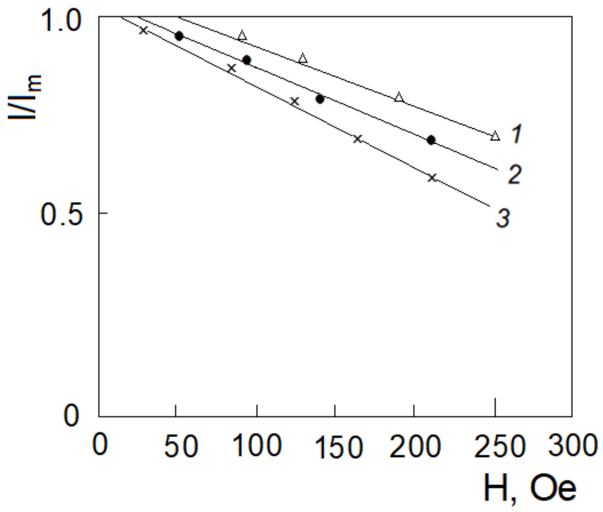

Fig. 5. Dependence of the normalized TPE intensity I/I_m on the MVP amplitude H: *1-3* at τ_m =1, 2 and 3 μs, respectively.

Analysis of the obtained results in Fig. 3 shows that in the case of cobalt the relation $A_m = H_0 \cdot \tau_m$=const, also holds according to which the MVP threshold area is constant for all MVP durations. This coincides with a similar conclusion for the MVP threshold area in the case of nickel ferrite, Fig. 1.

It should be noted that for the first time such relation was established in the NMR study of the pinning force H_0 of the ME signal dependence on τ_m [10] in cobalt. A_m is the threshold area of the MVP, corresponding to the beginning of the displacement of the DW under the action of the MVP. The physical meaning of this result could be understood if one takes into account that, according to the one-dimensional DW model [14], the DW displacement x under the action of a short MVP with amplitude H and duration τ_m is determined by the relation

$$x = C \cdot H \cdot \tau_m,$$

where C is a constant characteristic of the material under study. Thus, the same displacement of the DWs corresponds to the threshold value of the MVP, and it is natural to associate it with the width of the potential well in which the DW is located in the initial state.

We also note that a similar relationship between H_e and τ_m was established in the study of permalloy films by the Kerr magneto-optical method [15].

Let us further investigate the dependence of H_0 on the magnitude of the external magnetic field H_e at a fixed MVP duration τ_m =1 μs.

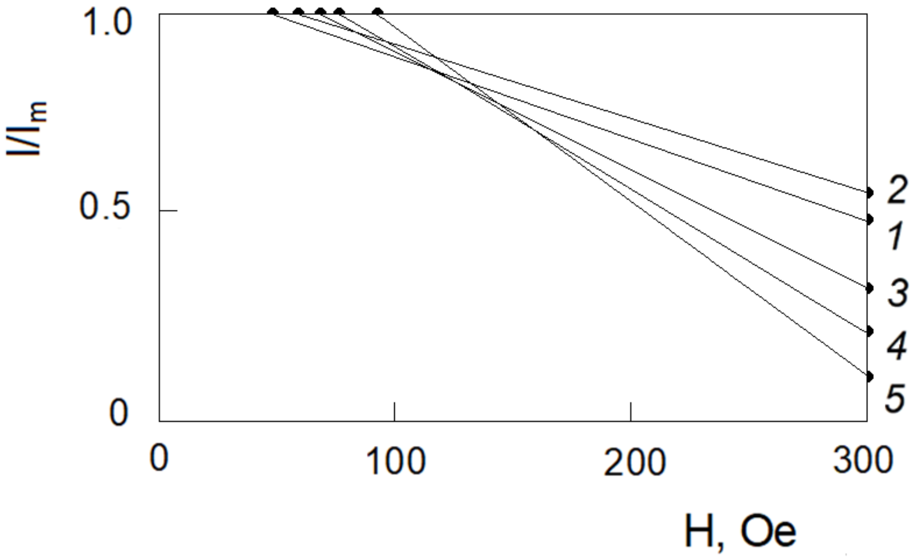

Fig. 6. Dependence of the normalized TPE amplitude I/I_m on the amplitude of the pulsed magnetic field at different (*1-5*) values of the external magnetic field H_e: 1-5: 0, 0.8, 1.9, 2.3, 3 kOe, respectively, at τ_m =1 μs.

Based on the data in Fig. 4, it is possible to construct the dependence of the pinning force H_0 on the magnitude of the external field H_e, Fig. 5.

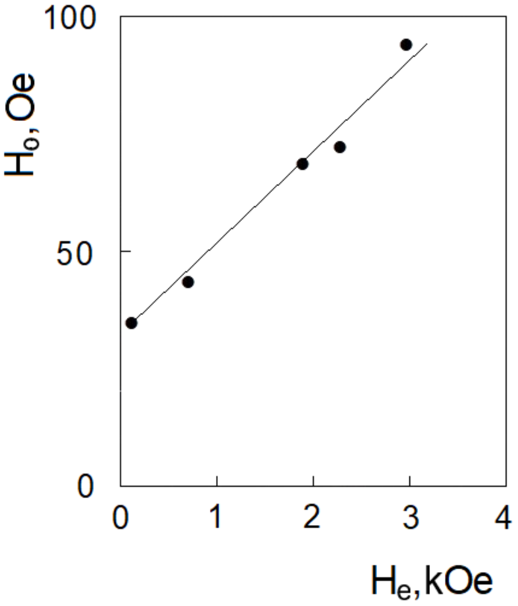

Fig. 7. Dependence of the pinning force H_0 on the magnitude of the external magnetic field H_e at τ_m =1 μs.

Thus, it has been established that up to external fields ~ 3 kOe, a linear dependence of H_0 on H_e is observed. This indicates that, at higher H_e, the DWs are distributed over positions corresponding to the parameters of the potential walls providing stronger pinning. We also note a feature of the data in Fig. 4, which consists in the fact that the rate of suppression of the TPE signal increases with increasing H_e, in contrast to that observed in lithium ferrite [6], which is due to the differences in the NMR properties of cobalt and lithium ferrite noted above [11].

The method of works [7,12] for the study of the effect of MVP on the two-pulse echo in samples of cobalt micro- and nanowires polarized in an external magnetic field in a paraffin matrix was used earlier in the work [5].

The results of the experiment are shown in Fig. 6 having the same character as for the micropowders studied in this work, i.e. they provide information about the pinning force and DW mobility in these samples.

Thus, it is shown in this work a possibility of studying the pinning force and mobility of DWs in micro- and nanowires using this powerful microscopic NMR method to characterize the magnetic properties of cobalt nanowires, along with the method of RF resonant magnetometry characterizing the bulk magnetic properties of samples under the study [5], Fig. 6

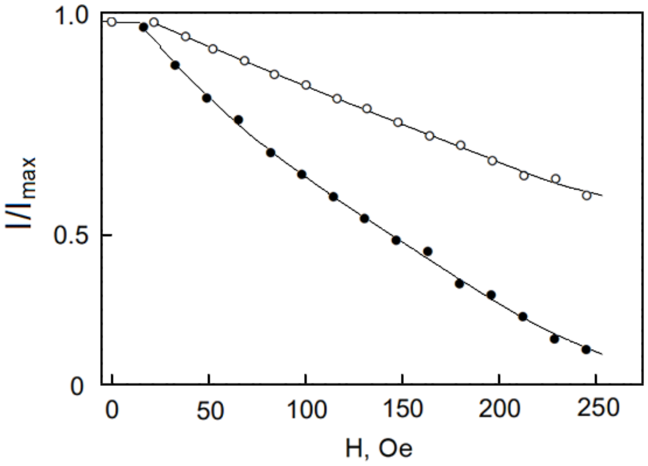

Fig. 8. MVP influence on the two-pulse echo intensity from nuclei arranged in the centers of DWs for cobalt microwires (● curve 1) and nanowires (○ curve 2), when MVP is directed in parallel to magnetic wires

Conclusion

In the present work, the pinning force of domain walls was studied by the NMR two-pulse spin-echo method in combination with an additional magnetic video-pulse in cobalt micro powders. It is shown that the pinning force is inversely proportional to the length of the magnetic video-pulse. In addition, the area of the magnetic video-pulse turned out to be constant for all threshold durations of the magnetic video-pulse.

The dependence of the pinning force H_0 on the external constant magnetic field H_e is studied. It is shown that up to ~3 kOe there is a linear dependence of H_0 on H_e. This indicates that as the H_e increases, the domain walls are distributed over sites corresponding to stronger pinning centers.

The comparative study of the two-pulse echo dependence on the magnetic video-pulse amplitude in cobalt micro- and nanowires was also carried out showing the opportunity to obtain microscopic information on the domain wall pinning by this method for cobalt nanowires

References:

[1] E.A. Turov, M.P. Petrov. Nuclear magnetic resonance in ferro and antiferromagnetics. John Wiley & Sons, Inc., Hoboken, New Jersey, 1972, 206 p.

[2] S. Wurmehl, J.T. Kohlhepp. “Nuclear magnetic resonance studies of materials for spintronic applications”, J. Phys. D: Appl. Phys. Vol. 41, pp. 173002 (2008).

[3] A.A. Shmyreva, V.V. Matveev, G.Y. Yurkov, “Nuclear magnetic resonance in magnetic nanomaterials as an effective technique to test and/or to certificate local magnetic properties”, Int. J. Nanotechnol., Vol. 13, pp. 126-135 (2016).

[4] G. Mamniashvili, M. Zviadadze, T.O. Gegechkori, Z.G. Shermadini, “**NMR spectroscopy of magnets using arbitrary number and duration radio-frequency pulses**”, Int. J. Trend Res. Dev. Vol. 3, pp. 434-473 (2016).

[5] G. Mamniashvili, Z. Shermadini, G. Donadze, T. Gegechkori, T. Zedginidze, T.Petriashvili, A.Peikrishvili, B.Godibadze, A.Maisuradze, “Electroless technology for production of cobalt

magnetic nanowires under magnetic fields for RE free magnet applications”, International.Scientific Journal “Air Transport”, No. 1(14), pp. 15-28 (2020).

[6] I.V. Pleshakov, P.S. Popov, Yu.I. Kuz’min, V.I. Dudkin, “NMR study of domain wall pinning in a magnetically ordered material”, Technical Physics Letters Vol. 42, No. 1, pp. 59-62 (2016).

[7] T. Gavasheli T. Gegechkori, G. Mamniashvili, G. Ghvedashvili, “NMR spin echo study of domain wall pinning in lithium ferrite in combination with an additional magnetic video-pulse”, Proceedings of XXVI International Seminar/Workshop on Direct and Inverse Problems of Electromagnetic and Acoustic Wave Theory (DIPED-2021), pp. 199-202 (2021).

[8] J.K. Galt, “Motion of individual domain walls in a nickel-iron ferrite”, Bell Syst. Tech. Vol. 33, pp. 1023-1054 (1954).

[9] L.A. Rassvetalov, A.B. Levitski, “ Influence of a pulsed magnetic field on the nuclear spin echo in some ferromagnets and ferrimagnets”, Sov. Phys. Solid State Phys. Vol. 23, No. 11. pp. 3354-3359 (1981).

[10] A.M. Akhalkatsi, G.I. Mamniashvili, T.I. Sanadze, "The nuclear spin-echo signals under combined action of magnetic field and RF pulses”, Appl. Magn. Res. Vol. 15, No. 3-4, pp. 393-399 (1998).

[11] G.I. Mamniashvili, T.O. Gegechkori, Ts.A. Gavasheli, “Study of the nature of the NMR signal in lithium ferrite upon exposure to a low-frequency magnetic field”, Phys. Met. Metallogr, Vol. 122, No. 9, pp. 841-846 (2021).

[12]T.A. Gavasheli, G.I. Mamniashvili, Z.G. Shermadini, T.I. Zedginidze, T.G. Petriashvili, T.O. Gegechkori, M.V. Janjalia, “Investigation of the pinning and mobility of domain walls in cobalt micro- and nanowires by the nuclear spin echo method under the additional influence of a magnetic video pulse”, J. Magn. Magn. Mater. Vol. 500, pp. 1555310 (2020).

[13] I.A. Kiliptari, V.I. Tsifrinovich, “Single-pulse nuclear spin echo in magnets”, Phys. Rev. B. Vol. 57, No. 18, pp. 11554-11564 (1998).

[14] S. Konishi, K. Mizuno, F. Watanabe, K. Narita, “Domain wall displacement under pulsed magnetic field”, AIP Conference Proceedings, Vol. 34, No. 1, pp. 145-147 (1976).

[15] D. Bartran, H. Bourne, “Domain wall velocity and interrupted pulse experiments”, IEEE Transactions on Magnetics vol. 9, No. 4, pp. 609-613, 1973.

იშვიათ მიწათა მეტალების არშემცველი მუდმივი მაგნიტების დომენური კედლების პინინგის შესწავლა ბირთვული მაგნიტური რეზონანსის მეთოდის გამოყენებით

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ანოტაცია

ნაშრომში განხილულია მოდიფიცირებული ბირთვული მაგნიტური რეზონანსის (ბმრ) ტექნიკის გამოყენების შესაძლებლობა კობალტის მიკროფხვნილებსა და ნანომავთულებში დომენური კედლის პინინგის (დაფიქსირების) შესასწავლად. კერძოდ, ინფორმაცია დომენის კედლის პინინგის შესახებ კობალტის ნანომავთულებში სასარგებლოა იშვიათ მიწათა მეტალების არშემცველი მუდმივი მაგნიტების მაგნიტური თვისებების ზუსტად დასარეგულირებლად.

კობალტის მიკროფხვნილებში ბირთვული მაგნიტური რეზონანსის მეთოდით დამატებითი მაგნიტური ვიდეოიმპულსის ერთობლივი მოქმედებისას პირველად იქნა შესწავლილი დომენური კედლების პინინგის ძალა, რომელიც დამოკიდებულია მაგნიტური ვიდეოიმპულსის ხანგრძლივობასა და მუდმივი მაგნიტური ველის მნიშვნელობაზე. ასევე ჩატარდა კობალტის მიკრო და ნანომავთულებში ორიმპულსიანი ექოს მაგნიტური ვიდეოპულსის ამპლიტუდაზე დამოკიდებულების შედარებითი კვლევა.

საკვანძო სიტყვები

ბირთვული მაგნიტური რეზონანსი; მაგნიტური ვიდეოიმპულსი; ორიმპულსიანი ექო; იშვიათ მიწათა მეტალების არშემცველი მუდმივი მაგნიტები; დომენური კედლის მობილურობა;