Inductive detection of magnetoacoustic responses and domain-acoustic echo in magnetostrictive materials using a pulse NMR technique

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The stimulated domain-acoustic echo having a long-time memory and the magnetoacoustic responses formed under excitation of magnetostrictive (ferrite, europium garnet, iron borate) and ferrite-piezoelectric layered composite samples by three radiofrequency (RF) pulses and by a train of single ones have been comparatively studied. These magnetoacoustic responses were inductively generated by placing a magnetostrictive sample into the RF coil of a pulse NMR spectrometer.

Проведены сравнительные исследования свойств обладающего долговременной памятью стимулированного домен-акустического эха и магнитоакустических откликов, формируемых при возбуждении магнитострикционных (феррит, европиевый гранат, борат железа) образцов и феррит-пьезоэлектрического слойного композита тремя или последовательностью единичных радиочастотных (РЧ) импульсов. Эти магнитоакустические сигналы индуктивно возбуждались путем помещения магнитострикционых образцов в резонансную РЧ катушку импульсного ЯМР спектрометра.

1. Introduction

Different type of magnetoacoustic responses were observed in [1, 2] after the excitation of a magnetostrictive sample mounted in RF coil of a conventional pulse NMR spectrometer by RF pulses. In [1], a slab shaped ferrite sample was excited by a train of RF pulses of 0.5 microseconds duration and 10 ms repetition time at 5 MHz frequency; a response signal was observed after each RF pulse. The Fourier transformation of that signal shows a series of equally spaced peaks with spaces depending on the sample geometry. Using these data for the slab shaped sample, one could find the acoustic wave speed in this material and compare with similar data obtained by other methods [3]. In [2], the domain-acoustic echo (DAE) signals were inductively generated using NMR spectrometer after application of three RF pulses in $Co_{0.01}Mn_{0.05}Cu_{0.18}Ni_{0.2}Mg_{0.72}Fe_{0.6}O_4$ ferrite, europium garnet and iron borate samples at 20 MHz frequency. The acoustic signal propagating in a ferrite sample after the application of the first RF pulse is recorded applying the second RF pulse of the same frequency. As a result of the simultaneous action of acoustic signal and the second RF pulse, a stationary space-periodic magnetic structure is formed in the sample with the period equal to the acoustic wave length. This structure is a magnetic image of the acoustic signal [4, 5].

The formation of this structure could be explained by the irreversible processes of magnetization change. The information storage duration is practically unlimited. The information is read by the third RF or the acoustic pulse as a DAE signal. In case of DAE, the irreversible change in magnetization could be caused by the displacement of domain walls in ferrite grains. In nanosized grains, this change in magnetization is due to magnetostriction [6]. The DAE phenomenon can be used to develop the DAE processors performing integral transformations of RF signals, memory devices, and delay lines [7].

The main properties of DAE were qualitatively well accounted for by a simple phenomenological model [6]. Unfortunately this model cannot be used in the direct quantitative comparison with the experimental results because of its simplicity. So far, polycrystalline ferrites with garnet or spinel structures have generally been used in DAE investigations of [8]. The main materials were iron-yttrium garnet $Y_3Fe_5O_{12}$ and nickel ferrite $NiFe_2O_4$. Various kinds of these ferrites ($Y_3Fe_{1.15}Al_{0.85}O_{12}$, $Ni_{0.98}Co_{0.02}Fe_2O_4$, $Ni_{0.97}Co_{0.03}Fe$ and $Ni_{0.97}Cu_{0.03}Fe_2O_4$) were used in DAE experiments. It is seen that in spinels, Fe^{2+} ions partially substituted by Co^{2+} and Cu^{2+} ones and in garnet, Fe^{3+} ions are partially substituted by Al^{3+} ones. The Co^{2+} and Cu^{2+} ions in, Ni–Co ($Ni_{1-x}Co_xFe_2O_4$) and Ni–Cu ($Ni_{1-x}Cu_xFe_2O_4$) ferrites, respectively, increase magnetoelastic interactions in these materials. Small admixtures of other ions could also change other physical properties of ferrites towards optimization of their use in the DAE processors. As an example, small aluminum admixtures in nickel ferrites change their saturation magnetization and a manganese admixture increases their electrical conductivity, while copper one could improve their mechanical properties, etc. As a result, the optimal ferrite composition could become rather complicated.

Additional experimental investigations are necessary to clear out the DAE formation mechanism in magnetostrictive materials with intense magnetoacoustic responses. With this purpose, we present here new results on DAE study in iron borate and ferrite-piezoelectric (FP) layered composite obtained using a conventional NMR spectrometer [9]. In a FP layered composite, the DAE and magnetoacoustic response signals were inductively excited and electrically detected. Besides, the comparative study of DAE properties and magnetoacoustic responses to action of a train of single RF pulses in slab shaped $Co_{0.01}Mn_{0.05}Cu_{0.18}Ni_{0.2}Mg_{0.72}Fe_{0.6}O_4$ ferrite, europium garnet, iron borate, and FP layered composite was carried out in this work using the same method [2]. DAE and magnetoacoustic response signals were for the first time inductively excited and electrically detected in FP layered composite samples, thus confirming their magnetoacoustic nature.

2. Experimental and results

The experimental setup used in this work to observe and investigate the DAE and magnetoacoustic signals comprised a standard Bruker Minispec p20 NMR spectrometer for studying the proton relaxation in liquids provided with a Kawasaki Electronica digital signal averager [2]. It was capable to produce 20 MHz RF pulses with durations and powers up to, correspondingly, 10 μs and 100 W. The DAE and magnetoacoustic response signals were observed and studied at room temperature in the rectangular sample ($6\times6\times6.3$ mm^3) of polycrystalline ferrite $Co_{0.01}Mn_{0.05}Cu_{0.18}Ni_{0.2}Mg_{0.72}Fe_{0.6}O_4$, europium garnet $Eu_3Fe_5O_{12}$ and iron borate $FeBO_3$ samples consisting of small disordered single crystals with the mean diameter about 1 mm placed in the cylindrical capsule of about 1 cm^3 volume. These signals were observed also under inductive excitation of FP layered composite samples consisting of five slab-shape ($10\times6\times1$ mm^3) layers of commercially available ferrite (F) material used as a ferrite core of television inductive coils and piezoelectric cerium titanium lead ceramics (P), forming PFPFP structure, and electrically detected by transferring electric signals from silver contacts connected to the two outer piezolayers to the receiver.

The NMR receiver was locked during action of RF pulse and strong transient response signal of RF coil (the receiver recovery time is denoted as 1 in Fig. 1 for DAE signal oscillograms). Any signal following this time moment was absent for the empty coil and for the coil with test manganese and lithium ferrite samples. The signals

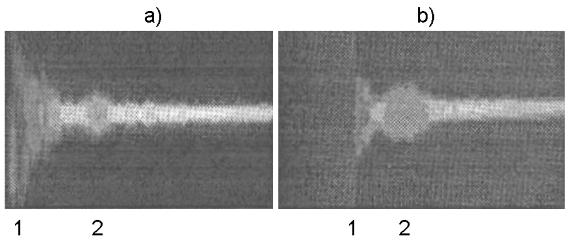

Fig. 1. Oscillograms of DAE signals in iron borate (*a*) and ferrite-piezoelectric layered composite (*b*). The time interval between the first two RF pulses τ_{12} = 20 μs; the RF pulse duration τ = 5 μs. The oscillograms were taken at room temperature and at external magnetic fields $H = 0$ (*a*, *b*). Total oscilloscope sweep duration is 100 μs. *1*, the third RF "read-out" pulse position; *2*, the DAE signal.

from samples under study were sufficiently intense providing their direct observation on oscilloscope without averaging. In addition, intense electric signals from piezoelectric layers were observed what clearly shows the magnetoacoustic nature of the observed signals.

The FP sample layers were cut using a diamond saw and stacked by epoxy.

In Fig. 1, the oscillograms of DAE signals in iron borate (*a*) and FP layered composite (*b*) samples are presented. The oscillograms were taken without detection. It was used the preamplifier of NMR spectrometer and a high-frequency oscilloscope with the frequency band 250 MHz. The DAE signals are similar to those in $Ni_{0.97}Cu_{0.3}Fe_2O_3$ ferrite [4]. In Fig. 2, the dependences of DAE amplitude on RF pulse power *P* are presented for the iron borate (*a*) and FP layered composite (*b*). The RF power was changed in the range from 3 to 50 W with the attenuator matched to 50 Ω. The RF power measurements were made directly by recording the RF pulse power on the excitation coil connected to the oscilloscope via the 50 Ω resistance.

As it is seen from the data obtained and results from [2], the power thresholds are observed within the range from ~2 W for europium garnet and up to ~15 W for iron borate. The dependences of DAE for ferrite and europium garnet on the external magnetic field created by a Helmholtz coil system were investigated in [2] and the corresponding result for the electrically detected DAE of the FP layered composite are presented in Fig. 3. The magnetic field was applied in parallel to the sample surfaces.

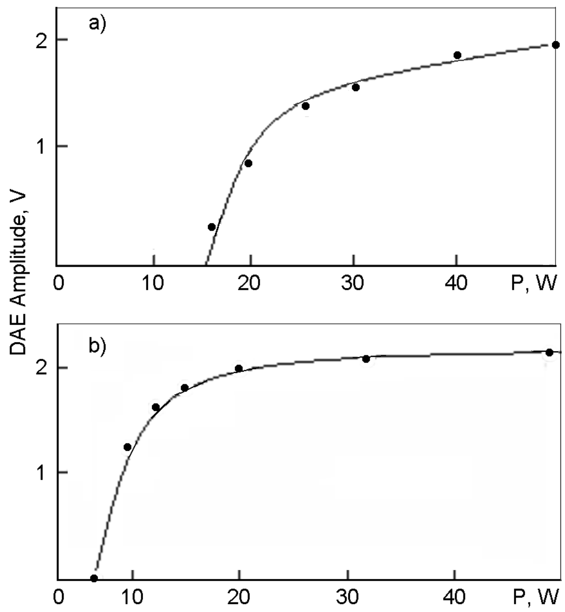

Fig. 2. The dependences of DAE amplitude on the RF pulse power in iron borate (*a*) and ferrite-piezoelectric layered composite (*b*).

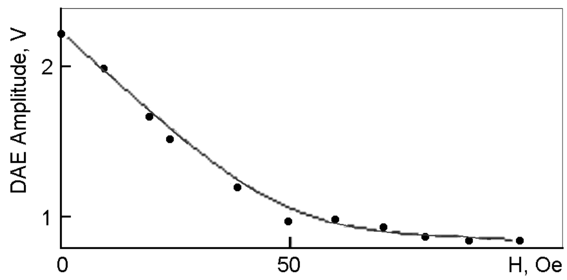

Fig. 3. The dependences of DAE amplitude on the external magnetic field strength for ferrite-piezoelectric layered composite.

The DAE are apparently observed only in the multidomain state in the external field up to ~200 Oe.

A low oscillating magnetic field at 100 Hz frequency and 100 Oe amplitude causes the suppression of DAE signals. This also points to the domain wall origin of the observed signals. As in [4], the DAE signals are characterized by anomalously large relaxation times.

The DAE signal amplitude recorded after the third "read-out" pulse decreases with the increase of the time interval between the first and the second "read-in" pulses. As an example, in the case of ferrite, this dependence shows an oscillating character (with a period ~4 μs) and full decay time ~22 μs.

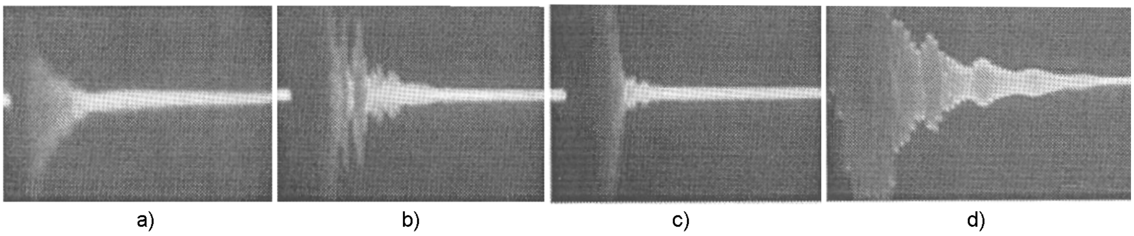

Fig. 4. Oscillograms of magnetoacoustic responses in $Co_{0.01}Mn_{0.05}Cu_{0.18}Ni_{0.2}Mg_{0.72}Fe_{0.6}O_4$ ferrite (*a*), europium garnet (*b*), iron borate (*c*) and ferrite-piezoelectric layered composite (*d*) under excitation by a train of RF pulses at the repetition rate $3 \cdot 10^3$ Hz and RF pulse duration 3 μs. Total oscilloscope sweep duration is 100 μs. Oscillograms were taken at zero external magnetic field and room temperature.

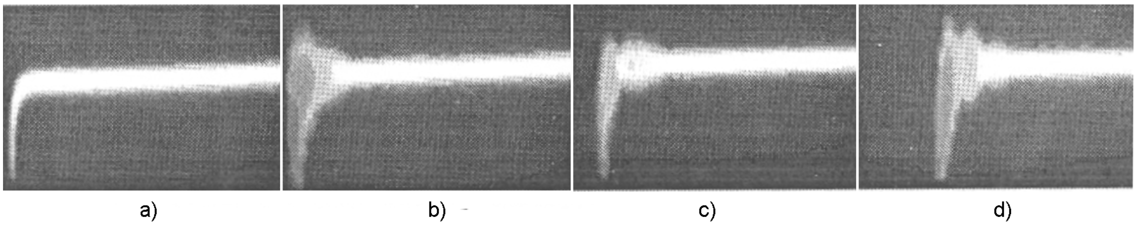

Fig. 5. Oscillograms of magnetoacoustic responses in $Co_{0.01}Mn_{0.05}Cu_{0.18}Ni_{0.2}Mg_{0.72}Fe_{0.6}O_4$ ferrite under excitation by a train of RF pulses at the repetition rate $3 \cdot 10^3$ Hz and RF pulse duration 2 μs (*a*), 3 μs (*b*), 6 μs (*c*) and 15 μs (*d*). Total oscilloscope sweep duration 100 μs. Oscillograms were taken at zero external magnetic field and room temperature.

The structurization in the DAE signal was also observed at increasing the time interval between the second and the third RF pulses. The DAE amplitude decreased at low temperatures and almost disappeared in a sample immersed in liquid nitrogen.

The DAE signal in iron borate had a more complex structure at higher magnetic fields. The main qualitative information is that its threshold RF power is ~15 W (Fig. 2a), its intensity increases significantly (by several tens of times) with the external magnetic field, reaching maximum at ~35 Oe, and it is suppressed by the external magnetic field around 140 Oe.

We present further the study results of magnetoacoustic responses generated by a train of single RF pulses taking as examples the measurements carried out on $Co_{0.01}Mn_{0.05}Cu_{0.18}Ni_{0.2}Mg_{0.72}Fe_{0.6}O_4$ ferrite, europium garnet, iron borate, and FP layered composite at the repetition rate $3 \cdot 10^3$ Hz and RF pulse duration 3 μs in the above described experimental conditions (Fig. 4). In Fig. 5, presented are response magnetoacoustic signals generated by a train of single RF pulses at the repetition rate $3 \cdot 10^3$ Hz and varying durations from 2 μs to 15 μs in $Co_{0.01}Mn_{0.05}Cu_{0.18}Ni_{0.2}Mg_{0.72}Fe_{0.6}O_4$ ferrite. At short durations, these signals are similar to those observed in [1]. It has been established also that the optimal RF pulse powers and magnetic fields for observation of these magnetoacoustic responses coincide with those for DAE signals.

3. Conclusion

The basic DAE and magnetoacoustic response signal properties in the studied materials mainly coincide with those observed in polycrystalline $Ni_{0.97}Cu_{0.03}Fe_2O_3$ ferrite [5] and in [1], respectively. The peculiarities could be apparently understood taking into account differences in shapes and physical parameters of samples, such as the coercitivity and mobility of domain walls. Further study in this direction is under way. Europium garnet is characterized by the most intense DAE signals at the lowest values of DAE RF excitation power threshold and external suppressing magnetic field. The DAE signal in iron borate has a much more complex structure in stronger external magnetic fields. Strong DAE signals were inductively excited and electrically detected in a ferrite-piezoelectric layered composite sample, showing clearly their magnetoacoustic nature.

The magnetoacoustic responses were observed in ferrite, europium garnet, iron borate, and ferrite-piezoelectric layered composite samples. Those have magnetostrictive nature common with DAE signals apparently related with domain wall displacements under the action of RF pulses in magnetostrictive materials.

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Індуктивне детектування магнітоакустичних відгуків та домен-акустичної луни у магнітострикційних матеріалах імпульсним ЯМР методом

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Виконано порівняльне дослідження стимульованої домен-акустичної луни з довгочасною пам'яттю та магнітоакустичних відгуків, що виникають при збудженні магнітострикційних зразків (ферит, європієвий гранат, борат заліза) та шаруватого ферит-п'єзоелектричного композиту трьома радіочастотними (РЧ) імпульсами та послідовністю поодиноких імпульсів. Ці магнітоакустичні відгуки індуктивно збуджувалися шляхом вміщення магнітострикційних зразків у резонансну РЧ котушку імпульсного ЯМР спектрометра.