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RESEARCH ARTICLE

Anisotropic magnetoelectric effect in lead zirconate titanate and magnetostrictive fiber composite structures

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Abstract
Objectives. The development of composite structures in which a strongly anisotropic magnetoelectric (ME) effect is observed is relevant for the creation of sensors that are sensitive to the direction of the magnetic field. Such an ME effect can arise due to the anisotropy of both the magnetic and the piezoelectric layers. In this work, a new anisotropic material named as a magnetostrictive fiber composite (MFC), comprising a set of nickel wires placed closely parallel to each other in one layer and immersed in a polymer matrix, is manufactured and studied. The study aimed to investigate the linear ME effect in a structure comprising of a new magnetic material, MFC, and lead zirconate titanate (PZT-19).
Methods. The magnetostriction for the MFC structure was measured using the strain-gauge method; the ME effect was determined by low-frequency magnetic field modulation.
Results. Structures with nickel wire diameters of 100, 150, and 200 μm were fabricated. The MFC magnetostriction field dependences were determined along with the frequency-, field-, and amplitude dependences of the ME voltage in the case of linear ME effect. Measurements were carried out at various values of the angle between the direction of the magnetic field and the wires. All samples demonstrated strong anisotropy with respect to the direction of the magnetic field. When the magnetic field orientation changes from parallel to perpendicular with respect to the nickel wire axes, the ME voltage decreases from its maximum value to zero.
Conclusions. The largest ME coefficient 1.71 V/(Oe · cm) was obtained for a structure made of MFC with a wire diameter of 150 μm. With increasing wire diameter, the resonance frequency increases from 3.5 to 6.5 kHz. The magnetostriction of the MFC is comparable in magnitude to that of a nickel plate having the same thickness.

Keywords: magnetoelectric effect, magnetostriction, fiber composites, piezoelectric effect, anisotropy

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НАУЧНАЯ СТАТЬЯ

Анизотропный магнитоэлектрический эффект в структуре цирконат–титанат свинца / магнитострикционный волоконный композит

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Резюме
Цели. Разработка композитных структур, в которых наблюдается сильно анизотропный магнитоэлектрический (МЭ) эффект, актуальна для создания датчиков, чувствительных к направлению магнитного поля. Такой МЭ эффект может быть обусловлен анизотропией как магнитного, так и пьезоэлектрического слоя. Авторами изготовлен новый анизотропный материал – магнитострикционный волоконный композит (МВК), представляющий собой набор никелевых проволок, расположенных вплотную параллельно друг к другу в один слой и погруженных в полимерную матрицу. Цель работы – исследование линейного МЭ эффекта в композитных структурах со слоями из МВК и керамики цирконата титаната свинца (ЦТС-19).
Методы. Магнитострикция МВК была измерена тензометрическим методом, МЭ эффект – методом низкочастотной модуляции магнитного поля.
Результаты. Были изготовлены структуры с диаметрами никелевых проволок 100, 150 и 200 мкм. Измерены полевые зависимости магнитострикции МВК, а также частотные, полевые и амплитудные зависимости МЭ напряжения для случая линейного МЭ эффекта при различной величине угла между направлением магнитного поля и проволоками. Показано, что все образцы обладают сильной анизотропией относительно направления магнитного поля. МЭ напряжение уменьшается от максимального значения до нуля при изменении направления магнитного поля с параллельного до перпендикулярного относительно волокон никеля.
Выводы. Наибольшим по величине МЭ коэффициентом, составляющим 1.71 В/(Э · см), обладает структура, изготовленная на основе МВК с диаметром проволоки 150 мкм. Частота резонанса растет от 3.5 кГц до 6.5 кГц с увеличением диаметра проволок. Величина магнитострикции МВК сопоставима по величине с магнитострикцией пластины никеля такой же толщины.

Ключевые слова: магнитоэлектрический эффект, магнитострикция, волоконные композиты, пьезоэффект, анизотропия

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INTRODUCTION

Magnetoelectric (ME) effects in layered composite structures containing ferromagnetic (FM) and piezoelectric (PE) layers realize mutual conversion of magnetic and electric fields. This in turn forms a basis for the creation of alternating and constant magnetic fields sensors, energy harvesting devices, controlled electronic devices (inductors and transformers), antennas, new types of magnetic memory, etc. [1–3]. In such composite structures, ME effects arise as a result of a combination of the magnetostriction of the FM layer and the piezoelectric effect in the PE layer [4]. When an ME structure is placed in an external magnetic field, the FM layer is deformed due to magnetostriction. Such deformations through the mechanical connection between the layers are transmitted to the piezoelectric layers, generating an electric field *e* due to the piezoelectric effect. In order to describe the efficiency of the ME effect, the corresponding coefficient $\alpha_E = e/h = u/(b \cdot h)$ was introduced, where b is the piezoelectric thickness; u is the electric voltage generated between the electrodes of the PE layer arising under the action of an alternating magnetic field h.

Particular attention was paid to the study of ME effects in isotropic layered composite structures, in which the magnitude of the effect does not depend on the direction of applied magnetic field H across the planar dimension of such structures. However, in some cases, for example, for magnetic field sensors that are sensitive to the direction of the magnetic field, the creation of anisotropic ME composites represents a topical problem, which can be solved either by means of FM layers with anisotropic magnetostriction or by means of the anisotropic piezoelectric effect occurring in PE layers. The anisotropic ME effect was observed in structures where a single crystal of cobalt ferrite $CoFe_2O_4$ [5] was used as a magnetic layer, while single crystals of lead magnioniobate titanate (PMN–PT) [6], lithium niobate $LiNbO_3$, or gallium phosphate $GaPO_4$ were used to form PE layers [7]. The anisotropy of magnetostriction can be artificially created during the fabrication of the structure. An anisotropic ME effect was also observed in heterostructures made from layers of piezoelectric ceramics of lead zirconate titanate (PZT, $Pb[Zr_xTi_{1-x}]O_3$, $0 \le x \le 1$) and FM ceramics $CoFe_2O_4$. Uniaxial magnetic anisotropy was created in the FM layer of such structures by applying external pressure to the layer during fabrication [8].

The anisotropy of the ME effect was also observed in structures that used piezo-fiber composites (PFC) manufactured by Smart Materials Corporation (Sarasota, Florida, USA) [9, 10]. PFCs comprise a set of rods made of piezoelectric ceramics arranged parallel to each other in a plane and placed in a polymer matrix. This material has become widespread due to the large size of the piezoelectric modulus, flexibility, and relatively low cost.

Recently, a new type of highly anisotropic magnetic material—magnetostrictive fiber composites (MFC)—has been proposed. This material comprises a set of wires made of a magnetic material (amorphous microwires or nickel) arranged parallel to each other at various distances and placed in a polymer matrix [11, 12]. The strongly anisotropic ME effect obtained by using this material as an FM layer in layered composites is due to the demagnetizing factor with respect to the direction of the external magnetic field (along the wires or across them).

In the present work, the authors fabricated several MFC samples based on nickel wires of different diameters and studied their magnetostrictive characteristics. The strongly anisotropic ME effect observed in MFC–PZT structures was analyzed in order to obtain the frequency-, field-, and angular dependences.

MATERIALS AND METHODS

Studies of the ME effect were carried out in two-layer composite structures containing layers of various MFCs and PZT piezoceramics. The MFC comprises a set of nickel wires closely arranged parallel to each other in a single layer and placed in a polymer matrix. Three MFC samples were made with wires of different diameters: 100 μm (sample 1), 150 μm (sample 2), and 200 μm (sample 3). BF-2 self-polymerizing adhesive based on formaldehyde resin was used as a matrix. The dimensions of the structures in the plane were 9.2 × 8.9 mm^2, 9.7 × 9.4 mm^2, and 8.4 × 8.3 mm^2, respectively. The MFC thicknesses were equal to the diameter of the wires. The appearance of the MFC is shown in Fig. 1a. The magnetostriction of all MFCs was ~$34 \cdot 10^{-6}$. When describing the results of measurements, subscripts 1, 2, and 3 are used in this work to denote MFCs having wire diameters of 100 μm, 150 μm, and 200 μm, respectively.

Commercially available $PbZr_{0.52}Ti_{0.48}O_3$ discs (Audiowell Electronics (Guangzhou) Co. Ltd., China) having a diameter of 16 mm and thickness of 200 μm were used as the PE layer. Silver electrodes were deposited on opposite sides of the disk and the disk was polarized in the transverse direction. Figure 1 presents photographs of the fabricated MFC and PZT–MFC structures. The layers of MFC and PZT were bonded

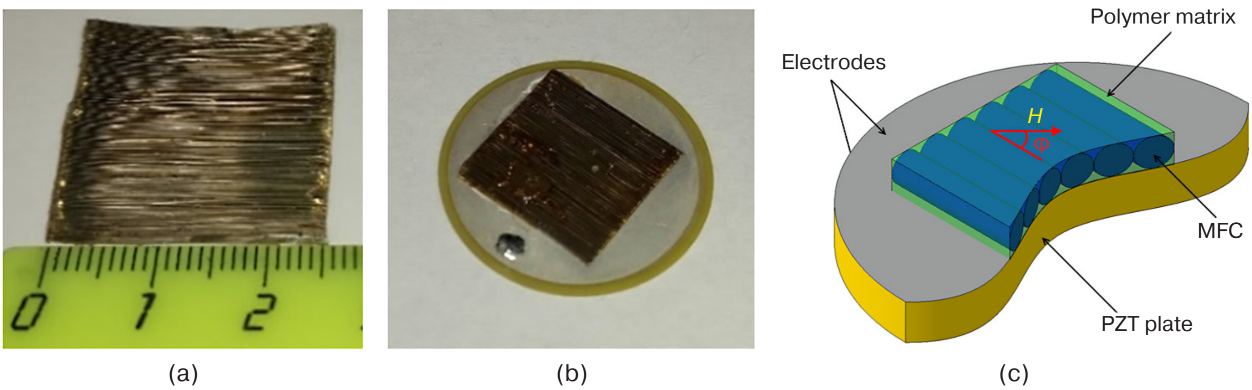

Fig. 1. Samples of MFCs (a), PZT–MFC with a diameter of 150 μm (b), and a schematic view of PZT–MFC structure (c)

using cyanoacrylate adhesive, while the structures were suspended in a magnetic field at two points located on opposite sides of the PZT disk using a special holder, allowing them to be rotated with respect to the direction of the magnetic field. According to the accepted classification of composites, the structures studied in the work belong to ME composites having a connectivity of “1–2” [13].

The ME effect in the structures was studied according to the method of low-frequency modulation of a magnetic field using the setup described in [12]. The structure was placed between the poles of an electromagnet in a constant magnetic field H in the range of ±1 kOe directed in the plane of the structure. The electromagnet was connected to a TDK Lambda GENH600-1.3 power supply (Japan). An exciting alternating magnetic field $h\cos(2\pi ft)$ with an amplitude of up to h = 0.8 Oe was created using Helmholtz coils connected to an Agilent 33210A arbitrary waveform generator (Agilent Technologies, Santa Clara, California, USA). The field h was parallel to the field H. The ME voltage $u(f)$ generated between the electrodes of the piezoceramic disk was measured using an AKIP 2401 voltmeter (China) with an input resistance of 10 MΩ at different values of f, h, φ, and H, as well as various orientations of H relative to the axis of the MFC wires. The dependences of the MFC magnetostriction on the magnetic field at different angles between the direction of the field and the direction of the fibers were measured by the tensometric method using the original setup described in [14].

RESULTS AND DISCUSSION

At first, the field and angular dependences of the MFC magnetostriction were studied. Figure 2 shows the field dependences of the MFC magnetostriction with different fiber diameters for cases when the magnetic field is directed along the wires. Within the measured range, it can be seen that samples 2 and 3

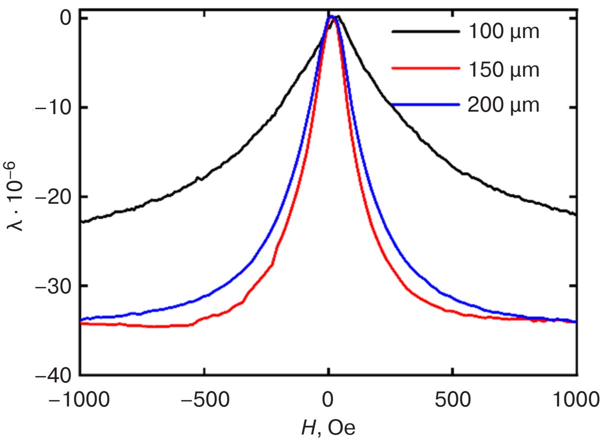

Fig. 2. Dependence of the magnetostriction for the fabricated MFC samples with different wire diameters on the magnetic field, H

reached saturation magnetostriction of ~(−34 · 10^{-6}), while sample 1 has a value of ~(−23 · 10^{-6}). Since the magnitude of the magnetostriction of sample 1 does not achieve saturation, it will be referred to as maximum in the text below. The saturation magnetic field decreases from more than 1 kOe to ~0.4 kOe as the fiber diameter increases from 100 μm to 200 μm. It can be seen that the magnitude of the magnetostriction of the MFC based on wires with a diameter of 150 μm increases faster than for a sample with a wire diameter of 200 μm. Here, the magnitude of the magnetostriction is apparently affected by both the demagnetizing factor and mechanical stresses from the matrix.

Figure 3 shows the measured dependences of the maximum magnetostriction on the angle between the direction of the magnetic field H and the nickel wires in fields up to 1 kOe. For all MFC samples, a strong anisotropy with respect to the direction of the magnetic field was observed. The maximum magnetostriction decreases almost to zero as the angle between the direction of the magnetic field and the wires increases to 90°. The dependence is symmetrical with respect to zero.

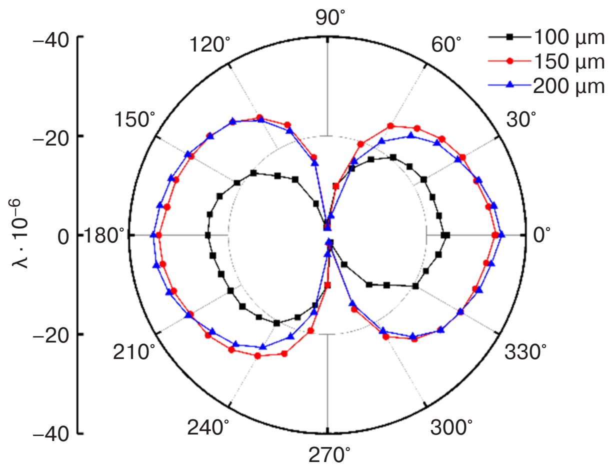

Fig. 3. Dependence of the MFC magnetostriction on the angle between the direction of the magnetic field and the direction of the wires for all samples

Next, measurements of the ME effect in the MFC–PZT structures were carried out. Figure 4 shows the frequency dependences of the ME voltage $u(f)$ obtained for all three structures. The measurements were carried out at h ~ 0.75 Oe and optimal values of the magnetic field strength H_m (H_m is the field in which the ME voltage value is maximum for each sample). The structures were magnetized along nickel wires.

The dependences show peaks at the frequencies of the bending resonance of the structures. For sample 1, the peak height was u_1 = 10.1 mV at a frequency $f_1 \approx 7.2$ kHz; for sample 2, u_2 = 25.9 mV at a frequency $f_2 \approx 7.9$ kHz; for sample 3, u_3 = 21.5 mV at a frequency $f_3 \approx 8.4$ kHz. It can be seen that the resonant frequency of the structures increases with an increase in the diameter of the MFC

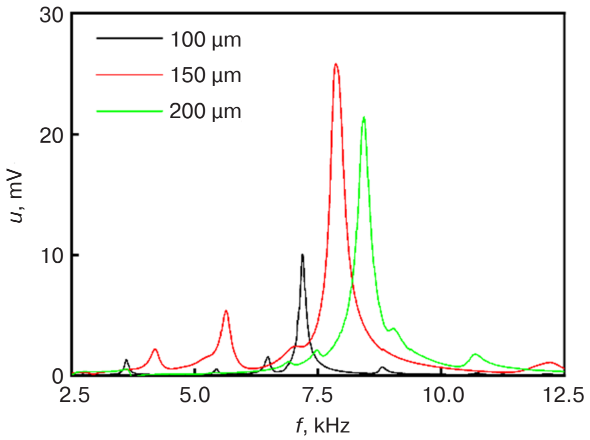

Fig. 4. Dependence of the ME voltage on the frequency measured in a magnetic field directed parallel to the wires

wires. The peak values are observed to increase linearly with an increase in the excitation field amplitude *h*. The quality factors of the resonances were $Q_1 \approx 72$, $Q_2 \approx 33$, $Q_3 \approx 32$. For each peak, the values of the ME coefficient were calculated using the formula $\alpha = u/(t \cdot h)$. Here *u* is the amplitude of the voltage generated by the ME structure; *t* is the thickness of the PZT layer; *h* is the amplitude of the alternating magnetic field. The calculated ME coefficients at resonant frequencies were $\alpha_1 = 0.61$ V/(Oe · cm), $\alpha_2 = 1.71$ V/(Oe · cm), and $\alpha_3 = 1.53$ V/(Oe · cm). While the obtained coefficients are comparable with the coefficients obtained in the PZT-nickel structures [15], they are significantly lower than the ME coefficients $\alpha_E \sim 10^2$ V/(Oe · cm) for the Metglas-AlN film structures [16]. The resonance parameters obtained from the data in Fig. 3 are given in Table.

Table. Parameters of resonant ME effect in MFC–PZT structures

Parameter	100 μm	150 μm	200 μm
ME voltage *u*, mV	10.1	25.9	21.5
ME coefficient α, V/(Oe · cm)	0.6	1.7	1.5
Resonance frequency *f*, kHz	7.2	7.9	8.4
Quality factor *Q*	72	33	32

Figure 5 shows the dependences of the ME voltage on the magnetic field *H* directed along the wires. The measurements were carried out at the corresponding resonant frequencies characteristic of each sample in an alternating magnetic field with an amplitude $h = 0.75$ Oe. For all samples, the dependences can be seen to have a typical form: the voltage initially increases with an increase in the field *H*, reaches a maximum in the field H_m corresponding to the maximum of the piezomagnetic coefficient ($q = d\lambda/dH|_H$), and then drops to almost zero when the magnetostriction is saturated. In the initial section, the field values H_m, which varied for each sample, were $H_{m1} \approx 70$ Oe, $H_{m2} \approx 50$ Oe, and $H_{m3} \approx 70$ Oe, respectively. All curves exhibited hysteresis. The maximum voltage amplitudes corresponded to the values shown in Fig. 4. The coercive fields H_c for samples 1–3 were $H_{c1} = 45$ Oe, $H_{c2} = 25$ Oe, and $H_{c3} = 35$ Oe, respectively.

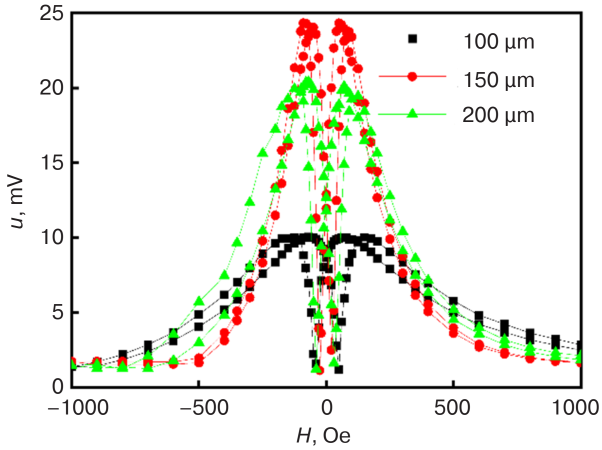

Fig. 5. Dependence of the ME voltage on the magnetic field *H* at the resonance frequency

At the next stage, we studied the dependences of the ME effect on the angle φ between the direction of the magnetic field and the axis of the wires ("angular dependences"), which was varied in the range from 0° to 360°. The measurements showed that, as this angle increases, the value of the maximum ME voltage decreases to practically reach zero at φ = 90°; this corresponds to the angular dependences of the magnetostriction shown in Fig. 3. The increase in the magnetic field H_m value at which the ME voltage reaches its maximum value, which is observed for each sample, is explained by the influence of the demagnetizing factor. Based on the obtained results, angular diagrams of the ME voltage in the field H_m at the resonance frequency are plotted

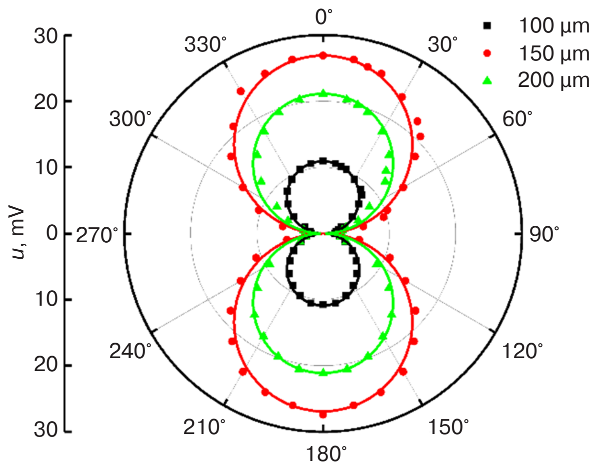

Fig. 6. Dependences of the ME voltage on the angle between the direction of the magnetic field and the axis of the wires

for all the studied samples (Fig. 6). It can be seen that the forms of dependences qualitatively coincide for all samples. The maximum voltage value was observed at the angle $\varphi = 0°$. With an increase in its value to 90° ME, the voltage decreased almost to zero. Thus, all samples demonstrate a strong anisotropy of the ME effect with respect to the direction of the magnetic field.

Figure 7 shows the dependences of the ME voltage on the amplitude of the alternating magnetic field, measured at the resonance frequency of the structures for the parallel orientation of the field H_m. Here, the dependences are linear over the entire range of amplitudes of alternating magnetic fields. The sensitivity of the structures to the magnetic field u/h was $u_1/h_1 \approx$ ≈ 12.4 mV/Oe for structure 1; $u_2/h_2 \approx 35.0$ mV/Oe for structure 2; $u_3/h_3 \approx 29.9$ mV/Oe for structure 3.

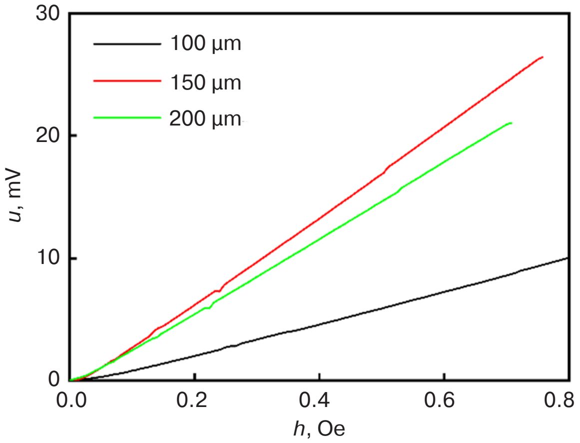

Fig. 7. Dependences of the ME voltage on the amplitude of the alternating magnetic field for samples based on MFC of various diameters

The frequencies of bending vibrations of a disk-shaped structure can be estimated using the following formula [17]:

$$f = k_{ns} \cdot \frac{a}{2\pi R^2}\sqrt{\frac{Y}{12\rho}}, \quad (1)$$

where k_{ns} is a constant, n is the number of nodal diameters, s is the number of nodal circles, a is the disk thickness, R is the disk radius, Y is Young's modulus, ρ is the density. Using the known values of the material parameters: $Y_m = 210$ GPa, $\rho_m = 8.9 \cdot 10^3$ kg/m^3, $Y_p = 59.5$ GPa, $\rho_m = 7.4 \cdot 10^3$ kg/m^3, and the dimensions of the structure, we obtain the frequencies $f_1 \approx 7$ kHz, $f_2 \approx 8.2$ kHz, and $f_3 \approx 8.3$ kHz. The frequencies determined in this way are in good agreement with the measured ones. Calculations showed that the resonant frequency increases with an increase in the diameter of the nickel wires, which also agrees with the measurements.

The amplitude of the ME voltage generated by the two-layer FM–PE structure in the linear mode is given by the formula [18, 19]:

$$u = AQ\frac{d_{31}}{\varepsilon}qh, \quad (2)$$

where A is the coefficient depending on the dimensions, mechanical and dielectric parameters of the layers of the structure, Q is the acoustic quality factor, d_{31} is the piezomodulus of the PE layer, $q = \lambda^{(1)} = \partial\lambda / \partial H|_H$ is the piezomagnetic coefficient, $\lambda(H)$ is the dependence of the magnetostriction of the FM layer on the magnetic field, and ε is the permittivity of the PE layer. It follows from formula (2) that the shape of the dependence of the ME voltage $u(H)$ on the constant magnetic field is largely determined by the field dependence $\lambda^{(1)}(H)$. Therefore, the shape of the angular dependence of the ME voltage coincides with the shape of the angular dependence of the MFC magnetostriction.

Figure 8 shows the dependences of the piezomagnetic modulus q calculated by the method of numerical differentiation for three MFC samples, which have been plotted according to the data of Fig. 2. The maxima of the piezomagnetic moduli were $q_1 \approx 0.06$ Oe^{-1}, $q_2 \approx$ ≈ 0.21 Oe^{-1}, and $q_3 \approx 0.17$ Oe^{-1} in fields of 60–80 Oe, which resulted in the maximum value of the ME voltage u being observed for this sample (see Fig. 4)

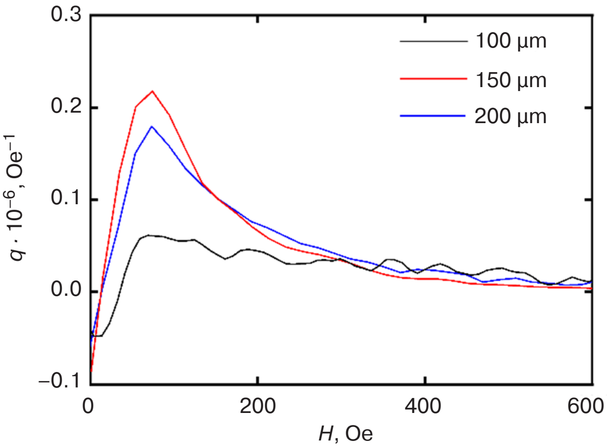

Fig. 8. Dependence of the piezomagnetic module of the MFC on the magnetic field for wires of different diameters

The anisotropy of the MFC magnetostriction is due to the demagnetizing factor. It is known that the field inside a ferromagnetic sample H_{in} is generally inhomogeneous and is related to the external field H as

$$H_{in} \approx H - N \cdot M(H_{in}), \quad (4)$$

where N is the demagnetizing factor depending on the shape of the sample and the direction of the field; M is the average magnetization of the sample, which depends on the field inside the FM layer H_{in} [20]. For a long nickel rod, when it is magnetized along the axis, the value is $N \approx 0$, and when it is magnetized across the axis, $N \approx 0.5$, i.e., when the nickel rod is magnetized along the axis, the field H_{in} is practically equal to H, and when the nickel rod is magnetized across the axis, the field H_{in} is much smaller than H. Since the value of magnetostriction λ is determined precisely by the internal field H_{in}, a much larger field should be applied in order to achieve the maximum coefficient $\lambda^{(1)}(H)$ when it is magnetized across the axis of the nickel rod. In the general case, it is also necessary to take into account the dipole-dipole interaction between the wires inside the matrix, which depends on the distance between them.

We note that structures of this kind have a great advantage over traditional magnetic materials, since the characteristics of their ME effects can be controlled by changing the distance between the MFC wires, as well as their diameter and the materials used in the wires and matrix. These issues require further research.

CONCLUSIONS

Thus, a linear ME effect in MFC–PZT two-layer composite structures was determined and analyzed. The MFC samples comprised a set of nickel wires of different diameters arranged parallel to each other in a single layer in a polymer matrix. The frequency and field characteristics of structures with wires 100–200 μm in diameter were measured at the flexural resonance frequency. It was shown that the resonance frequency increases from 7.2 kHz to 8.4 kHz with increasing wire diameter. The highest ME coefficient of 1.7 V/(Oe · cm) obtained for a sample with a nickel wire diameter of 150 μm is comparable with the ME coefficient obtained for a structure of comparable thickness with an FM layer of polycrystalline nickel. The magnitude of the ME effect strongly depends on the orientation of the constant magnetic field H due to the anisotropy of the MFC magnetostriction.

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Authors' contributions

D.V. Saveliev—description of the research methodology, preparing test samples and installations, conducting research, data curation, visualization, and writing the text of the article.

L.Y. Fetisov—conceptualization, description of the research methodology, preparing test samples and installations, conducting research, validation, and writing the text of the article.

V.I. Musatov—preparing test samples and installations, conducting research, data curation, writing the text of the article.

M.V. Dzhaparidze—preparing test samples and installations and conducting research.

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