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CoFe/Cu/CoFe/FeMn spin valves and CoFe/Cu/CoFe three-layer nanostructures at microwave frequencies

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Received June 24, 2022
Revised July 10, 2022
Accepted July 21, 2022

The microwave magnetoresistance of CoFe/Cu/CoFe/FeMn spin valves and CoFe/Cu/CoFe three-layer nanostructures with high magnetoresistance has been studied. The transmission and reflection coefficients were measured at the frequency range from 26 to 38 GHz in magnetic fields up to 12 kOe. It is shown that the dependences of the transmission coefficient of spin valves are not symmetric with respect to the $H = 0$ axis, as well as the dependences of magnetoresistance. It is established that the relative changes in the microwave transmission coefficient are 1.5−2 times higher than the relative magnetoresistance measured at direct current. Changes in the reflection coefficient have a smaller value and the opposite sign with respect to changes in the transmission coefficient.

Keywords: metal superlattices, spin valves, ferromagnetic resonance, ferromagnetic antiresonance, microwave giant magnetoresistance effect.

DOI: 10.21883/TP.2022.11.55179.168-22

Introduction

A characteristic feature of the „spin valve" type nanostructures is a sharp change in their electrical resistance in the area of weak magnetic fields. Compared to superlattices, spin valves have a higher sensitivity to the magnetic field. Because of this property, spin valves are used as sensing elements in sensors and readout heads [1], in various applications of magneto electronics and spintronics [2–8]. In spin valves in relatively weak fields, one of the ferromagnetic layers, called the free layer, is re-magnetized. The composition of the spin valves includes a second ferromagnetic layer, fixed by exchange interaction with a neighboring layer of antiferromagnetic material. Between the free and fixed ferromagnetic layers in the spin valve is a non-magnetic layer. Important functional characteristics of spin valves are the magnitude and sign of the switching field as well as the hysteresis value. The hysteresis in the magnetization and magnetoresistance of the spin valves was investigated theoretically in [9]. The dependences of the hysteresis loop width on the value of the exchange field acting on the free ferromagnetic layer were obtained. The effect of magneto-dipole interaction on the equilibrium magnetic state of three-layer nanostructures and on the dynamics of magnetic moment under the action of a pulse of sub-nanosecond duration was studied in [10]. The properties of the antiferromagnetic material layer determine the thermal stability and set the upper temperature limit to which the spin valves retain their properties [11]. The giant magnetoresistance of Co/Cu/Co spin valves was calculated ab initio [12]. The exchange interaction in three-layer CoFe/Cu/NiFe nanostructures as a function of Cu spacer thickness was investigated in [13] using the ferromagnetic resonance method. Giant magnetoresistance enhancement in epitaxial multilayer $Co_{50}Fe_{50}$/Cu nanostructures with a metastable spacer of bulk-centered copper is found in [14]. The CoFe/Cu system is attractive for sensor applications due to its high magnetoresistance and small hysteresis loop width, obtained, for example, by using a compound buffer layer Ta/$(Ni_{80}Fe_{20})_{60}Cr_{40}$ =Ta/PyCr, contributing to the sharp $\langle 111 \rangle$ texture. The preparation of spin valves with optimal characteristics requires consideration of magnetic anisotropy, requires careful material selection, buffer layer thicknesses, and thermomagnetic treatment [15].

The study of the giant magnetoresistive effect (GMR) on microwaves allows us to determine the frequency dependence and existence limits of the effect, as well as the joint effect of ferromagnetic resonance and GMR on the measured quantities. The current state of research of magnetic metallic nanostructures by methods of microwave transmission and reflection is analyzed in [16]. The microwave giant magnetoresistive effect (μGMR) in spin valves has been studied in [17]. There, a μGMR study of electromagnetic wave propagation and reflection from Ta(10)/NiFe(3)/IrMn(6)/CoFe(1.5−3)/Cu(2.5)/CoFe(1)/NiFe(2)/Ta(2) spin flaps is performed, here the layer thickness in nanometers is in parentheses. In [17] it is shown that the field dependence of the reflectance, in comparison with the dependence of the transmittance coefficient, has the opposite sign and a smaller value.

This paper presents the results of studies of the frequency characteristics of the microwave magnetoresistance of spin valves and three-layer nanostructures of the CoFe/Cu/CoFe system. The investigated spin valves have a maximum magnetoresistance of $\sim 10-12\%$ and magnetoresistance sensitivity of about 0.3%/Oe. Microwave measurements are made by the method of transmission and reflection at frequencies of the millimeter range. Changes in the coefficient of passage and giant magnetoresistance of spin flaps were compared. The microwave properties of CoFe/Cu/CoFe/FeMn spin valves and CoFe/Cu/CoFe triplet nanostructures are compared. The purpose of this comparison is to reveal the effect of the FeMn antiferromagnetic layer on the re-magnetization processes of the layers and on the microwave characteristics.

1. Preparation and X-ray examination of samples

The material, thickness and microstructure features of each layer, and magnetic anisotropy induced by sputtering or subsequent thermomagnetic treatment are important for obtaining the required magnetoresistance characteristics of the spin valve. The choice of materials for the buffer layer of spin valves is devoted to works [18–20]. It is known that the formation of the $\langle 111 \rangle$ texture in spin flaps based on FCC of materials allows one to significantly reduce the width of the hysteresis loop, which is explained by a decrease in local fluctuations of magnetic crystal anisotropy in the film plane.

Spin valves were produced by magnetron sputtering using the MPS-4000-C6 high-vacuum magnetron sputtering machine (Ulvac). Sputtering is performed in an atmosphere of extremely pure argon at a pressure of 0.1 Pa. The substrate is rotated during sputtering to ensure uniform film thickness across the entire surface of the substrate. The main process parameters for sputtering nanostructures are as follows: magnetron power — 100 W; substrate rotation frequency — 8 rpm; magnetic field strength in the substrate plane — 80 Oe; substrate temperature during sputtering — room temperature. Determination of the sputtering speed of each material was performed on the prepared auxiliary two-layer films with a step by measuring the step height with an optical profilometer-interferometer Zygo NewView 7300. For the $Co_{90}Fe_{10}$ alloy the sputtering rate was 2.7 nm/min, for Cu — 6.5 nm/min.

Samples of the following compositions were grown: spin valves glass/Ta(5)/PyCr(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/FeMn(15)/Ta(5) — sample № 1, glass/Ta(5)/PyCr(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/FeMn(20)/Ta(5) — sample № 2; three-layer nanostructures glass/Ta(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/Ta(5) — sample № 3, glass/Ta(10)/CoFe(3.5)/Cu(2)/CoFe(3.5)/Ta(5) — sample № 4. The samples were grown on a 0.2 mm thick Cornig glass substrate. The samples of spin valves differ from each other by the thickness of the FeMn antiferromagnetic layer, and the samples of three-layer nanostructures — by the thickness of the tantalum buffer layer. X-ray studies were performed on a PANalytical Empyrean diffractometer in Co radiation with a wavelength of 1.79 Å in a parallel beam geometry and a plane-parallel collimator with a flat graphite monochromator. The quality of the layered structure, the thicknesses of the layers and the degree of imperfection of the interlayer boundaries of the samples were determined on the basis of X-ray reflectometry data. Reflectograms were processed using the PANalytical X'Pert Reflectivity program.

Figure 1, *a* shows the reflection patterns for the samples glass/Ta(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/Ta(5) (sample № 3), glass/Ta(10)/CoFe(3.5)/Cu(2)/CoFe(3.5)/Ta(5) (sample № 4). All samples have a lamellar structure, sharp interlayer boundaries with an average roughness $3-7$ Å. The X-ray diagram for the glass/Ta(5)/PyCr(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/FeMn(20)/Ta(5) spin valve (sample № 2) in the low angle area is shown in Figure 1, *b*. The difference between the measured radiograph and the result of its processing is noticeable only in the interval of angles $6-8^\circ$, and in general a good correspondence of the dependences is obtained. This proves the presence of a lamellar structure and sharp interlayer boundaries.

2. Magnetic and magneto-resistive properties

When measuring the magnetic characteristics of spin valves, highly sensitive measuring equipment is required due to the small amount of magnetic material in the sample. The sharp change in magnetization occurs in the area of weak magnetic fields with a strength of several oesteds to tens of oesteds, so the main results were obtained using a specially designed automated vibration magnetometer. The magnetometer has the following specifications: maximum magnetic field strength: ± 20 kOe; error of measurement of magnetic moment 3%. Magnetoresistance was measured by the four-contact method in fields up to ± 2 kOe. The magnetic field was directed in the plane of the layers perpendicular to the current density vector. Magnetization and magnetoresistance measurements were performed at room temperature.

Consider the change in the orientation of the magnetic moments of the layers during the remagnetization of the spin flaps. The magnetic moment of one ferromagnetic layer is anchored by exchange interaction with the neighboring antiferromagnetic layer. The remagnetization of this layer occurs in fields close to the field of exchange H_{EX} displacement, determined by the exchange interaction at the ferromagnetic/antiferromagnetic boundary. The second ferromagnetic layer is jump-magnetized in a switching field close to $H = 0$ [15]. The magnitude of the switching field H_I is determined by the magnitude of the interaction between the free and fixed layers. In the interval of fields between

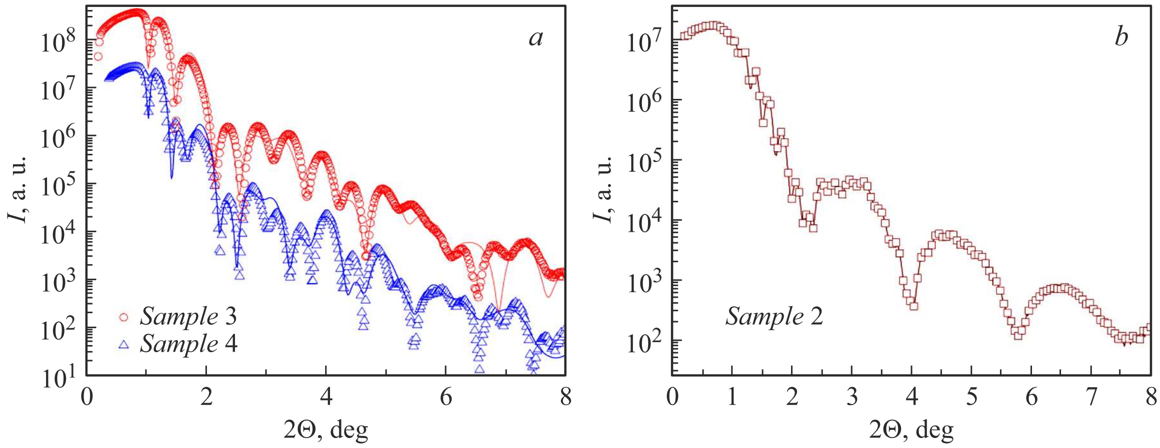

Figure 1. Experimental data (symbols) and processing result (lines) of X-ray reflectometry for three-layer nanostructures (*a*); X-ray diffractogram for spin valve sample № 2 in the low angle area (*b*).

H_I and H_{EX} an antiferromagnetic ordering of the magnetic moments of the layers is established. Unidirectional anisotropy leads to a shift in the remagnetization loop of the fixed layer by the value of the exchange displacement field. Unidirectional anisotropy is formed during sputtering of the nanostructure in a magnetic field. The direction of the unidirectional anisotropy axis coincides with the direction of the magnetic moment of the adjacent ferromagnetic layer when the nanostructure is sputtered and can be changed by special thermomagnetic treatment [15].

The magnetic hysteresis loop for the glass/Ta(5)/PyCr(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/FeMn(20)/Ta(5) — spin valve sample is shown in Fig. 2, *c*. In intervals of fields less than –20 Oe and more than 200 Oe a parallel orientation of the magnetic moments of the two CoFe layers is realized. It can be seen that for the spin valve the loop symmetry is not carried out, in Fig. 2, *c* the loop is shifted to the area of positive fields. A similar hysteresis loop is observed in the sample № 1.

A four-contact method was used to measure the magnetoresistance of film samples. The magnetoresistance dependences for the samples of three-layer nanostructures № 3 — glass/Ta(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/Ta(5) and № 4 — glass/Ta(10)/CoFe(3.5)/Cu(2)/CoFe(3.5)/Ta(5) are shown in Fig. 3. Over the entire range of magnetic field to field from to, a smooth change in resistance is observed, depending on the angle between the magnetizations of the layers. The maximum value of the relative magnetoresistance of the sample № 3 with a buffer layer thickness of 5 nm is slightly higher than that of the sample № 4.

The magnetoresistance dependences for spin valves look somewhat different № 1 — glass/Ta(5)/PyCr(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/FeMn(15)/Ta(5) and № 2 — glass/Ta(5)/PyCr(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/FeMn(20)/Ta(5). In the switching field near $H = 0$ there is a sharp change in resistance in a narrow range of weak magnetic fields (Fig. 4). There is hysteresis in the magnetoresistance dependence. For sensor applications, it is important that this hysteresis in the small-field area is of minimum width. Magnetoresistive dependences of spin valves have no symmetry with respect to the $H = 0$ axis.

3. Microwave transmission and reflection

The methodology for microwave transmission and reflection measurements is described in [16,21,22]. The sample is placed in a metal mandrel, and the edges are thoroughly coated with conductive paste to prevent unwanted seepage of waves besides the sample. The mandrel with the sample is placed between the flanges of the rectangular waveguide. Thus, the sample completely overlaps the cross section of the waveguide. The transmission T and reflection R modulus and their changes in the magnetic field are measured. The relative changes in the coefficients are defined as $t_m = \big(|T(H)| - |T(0)|\big)/|T(0)|$, where $|T(H)|$ — the modulus of the transmittance and $r_m = \big(|R(H)| - |R(0)|\big)/|R(0)|$, where $|R(H)|$ — the modulus of the reflectance.

Fig. 5 shows the magnetic field dependence of the microwave coefficients for a sample of three-layer nanostructure № 4 — glass/Ta(10)/CoFe(3.5)/Cu(2)/CoFe(3.5)/Ta(5). Fig. 5, *a* shows the results of the transmittance measurements at several frequencies, showing the weak frequency dependence of μGMR. Fig. 5, *b* compares the dependencies of the transmission and reflectances. The changes in the transmittance factor have a positive sign, and the reflectance — a negative sign, and these changes are smaller in magnitude. The changes in the coefficients in the magnetic field for three-layer nanostructures are symmetrical with respect to the $H = 0$ line.

In measurements of the three-layer № 3 — glass/Ta(10)/CoFe(3.5)/Cu(2)/CoFe(3.5)/Ta(5) nanostructure, a ferromagnetic resonance (FMR) was observed besides μGMR (Fig. 6, *a*). Changes in weak fields of the order of ± 0.3 kOe are caused by μGMR, similar to the

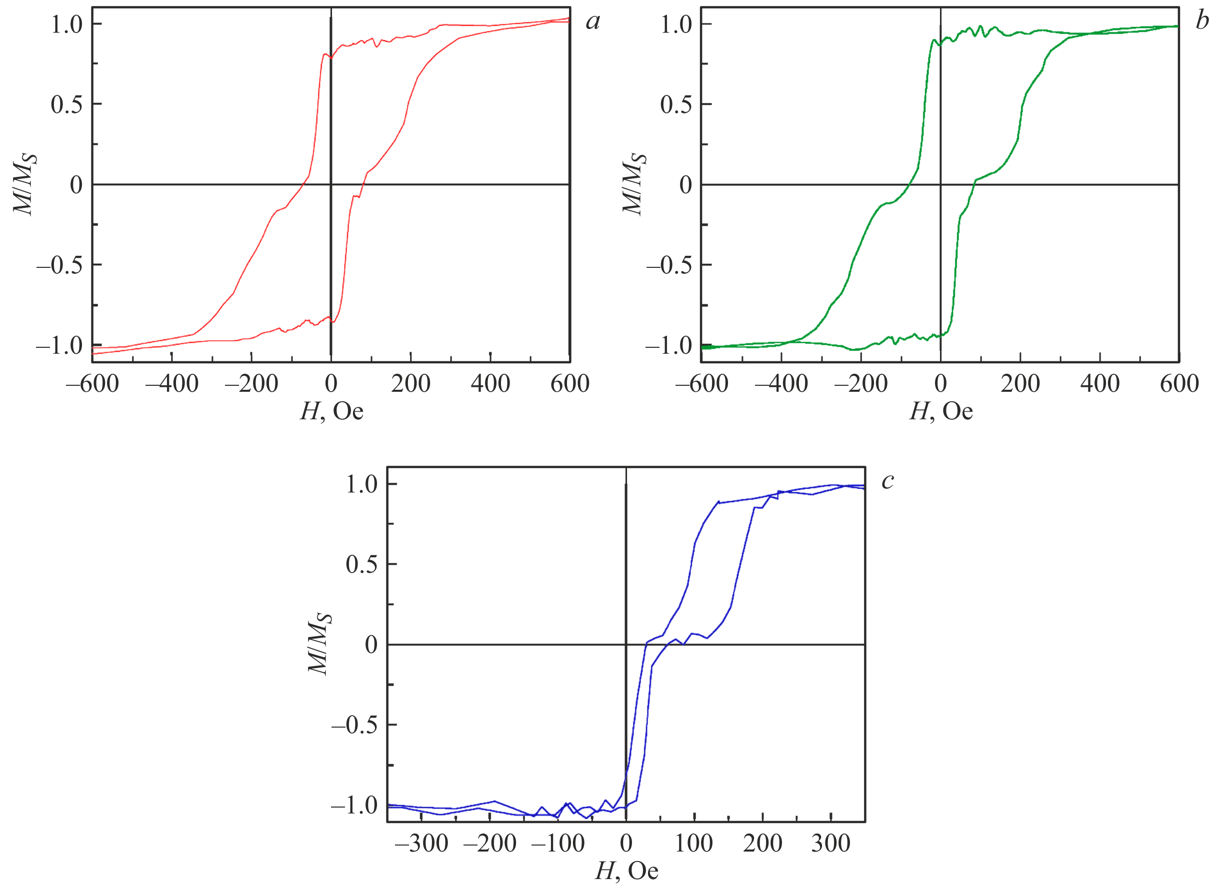

Figure 2. Hysteresis loops measured at room temperature in glass/Ta(5)/CoFe(3.5)/Cu(2)/CoFe(3.5)/Ta(5) (sample № 3) (*a*), glass/Ta(10)/CoFe(3. 5)/Cu(2)/CoFe(3.5)/Ta(5) (sample № 4) (*b*); magnetic hysteresis loop for sample № 2 — spin valve composition glass/Ta(5)/PyCr(5)/CoFe(3. 5)/Cu(2)/CoFe(3.5)/ FeMn(20)/Ta(5) (*c*).

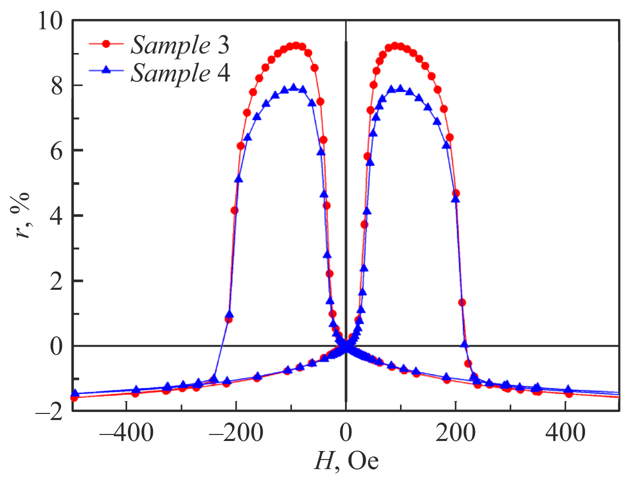

Figure 3. Magneto-resistivity dependences for samples of three-layer № 3 and № 4 nanostructures.

sample № 4. The minimum of the transmission coefficient in the fields ~ 8 kOe is caused by wave absorption when the FMR condition is met. Figure 6, *b* shows the dependencies measured in the field interval from 2 to 12 kOe at several frequencies. On the dependences measured at frequencies 26, 29 and 32 GHz, there is a minimum due to FMR. The minimum field shifts to a area of stronger fields with increasing frequency, and at 35 and 38 GHz a field of 12 kOe is not enough to reach the minimum. At frequencies of 32 GHz or more in fields smaller than the FMR field, there is a maximum of the transmittance factor. As indicated in [23], this maximum is caused by ferromagnetic antiresonance (FMAR), which in ferromagnetic metal films and nanostructures corresponds to a field in which the real part of the effective magnetic permeability turns to zero. The FMR and FMAR spectra (Fig. 6, *c*) are plotted according to the position of the extrema in Fig. 6, *b*. Three-layer nanostructures have previously been little investigated by wave transmission and reflection [18] methods. Compared to the work [18], the present work measured the field dependences not only of the transmission coefficient, but also of the reflectance, and investigated the FMAR effect.

The μGMR effect is observed in spin valve samples at frequencies in the millimeter wavelength range. These are higher frequencies than those used in [17]. The measurement results for the sample № 1 are shown in

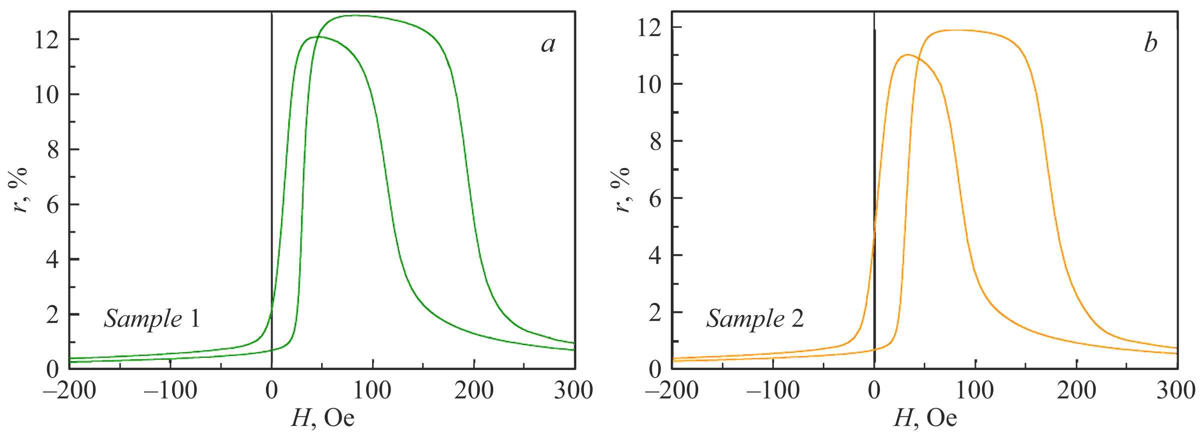

Figure 4. Magnetoresistance dependences for spin valve samples № 1 (*a*) and № 2 (*b*).

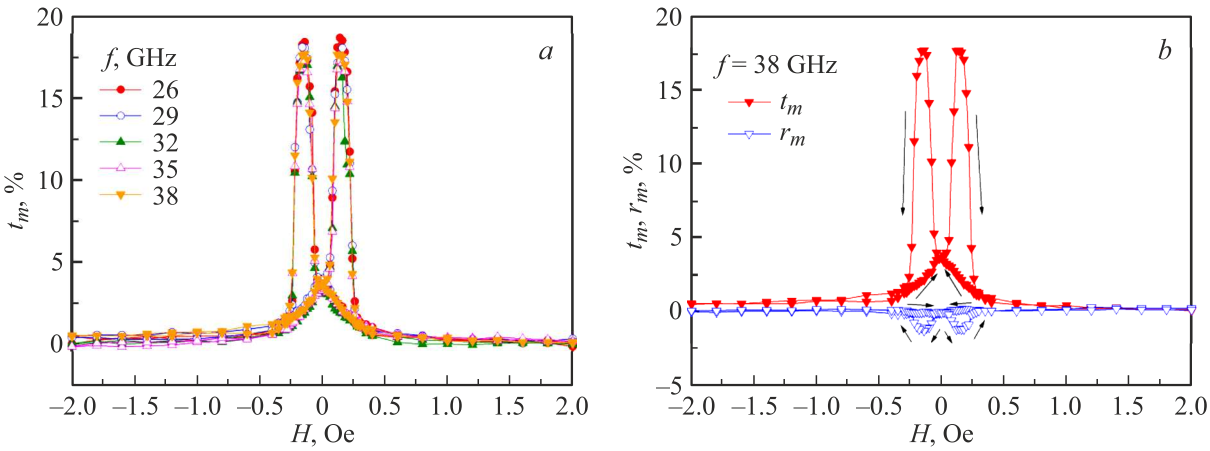

Figure 5. Magnetic field dependences of microwave coefficients for a sample of three-layer № 4 nanostructure: measurements of transmittance at several frequencies (*a*); comparison of transmission and reflectance dependences (*b*).

Fig. 7. Fig. 7, *a* shows the magnetic field dependence of the microwave transmission coefficient at several frequencies. It can be seen that the dependencies measured at different frequencies are close to each other, so that the frequency dependence of the μGMR effect is weak. The dependence of the reflectance at the frequency $f = 26$ GHz is shown in Fig. 7, *b*. As in the case of three-layer nanostructures, the field dependence of the reflectance has a negative sign and is much smaller in magnitude than the dependence of the transmission coefficient. The field dependences of the transmission coefficients and reflectances of microwaves for spin flaps have no symmetry with respect to the $H = 0$ line.

4. Discussion of results

Let us compare the magnetoresistance and changes in the microwave transmittance. It follows from earlier work [16] that in the approximation in which the multilayer nanostructure is replaced by a homogeneous film with effective parameters, the relative magnetoresistance and relative changes in the microwave transmission coefficient are approximately equal for the centimeter and millimeter wave ranges. This equality follows from the formula for the coefficient of passage T of an electromagnetic wave through a metal plate

$$T = \frac{2Z_m}{2Z_m \operatorname{ch} k_m d + Z \operatorname{sh} k_m d}, \quad (1)$$

which is a special case of the flat layer reflectance expression [24,25] for the case of a well-conducting medium.

In formula (1) $k_m = (1 + i)/\delta$ — the wave number under normal skin effect, δ — the skin layer depth, d — the total metal thickness of the entire nanostructure. The impedance of the metallic nanostructure Z_m is much smaller than the impedance of the waveguide Z, $|Z_m| \ll Z$. At the millimeter range waves the inequality of $d \ll \delta$ is carried out. The inequality $k_m d \ll 1$ is fulfilled for thickness values of nanostructures from units to hundreds of nanometers, and from formula (1) follows a one-to-one correspondence between changes in the permeation coefficient t_m and

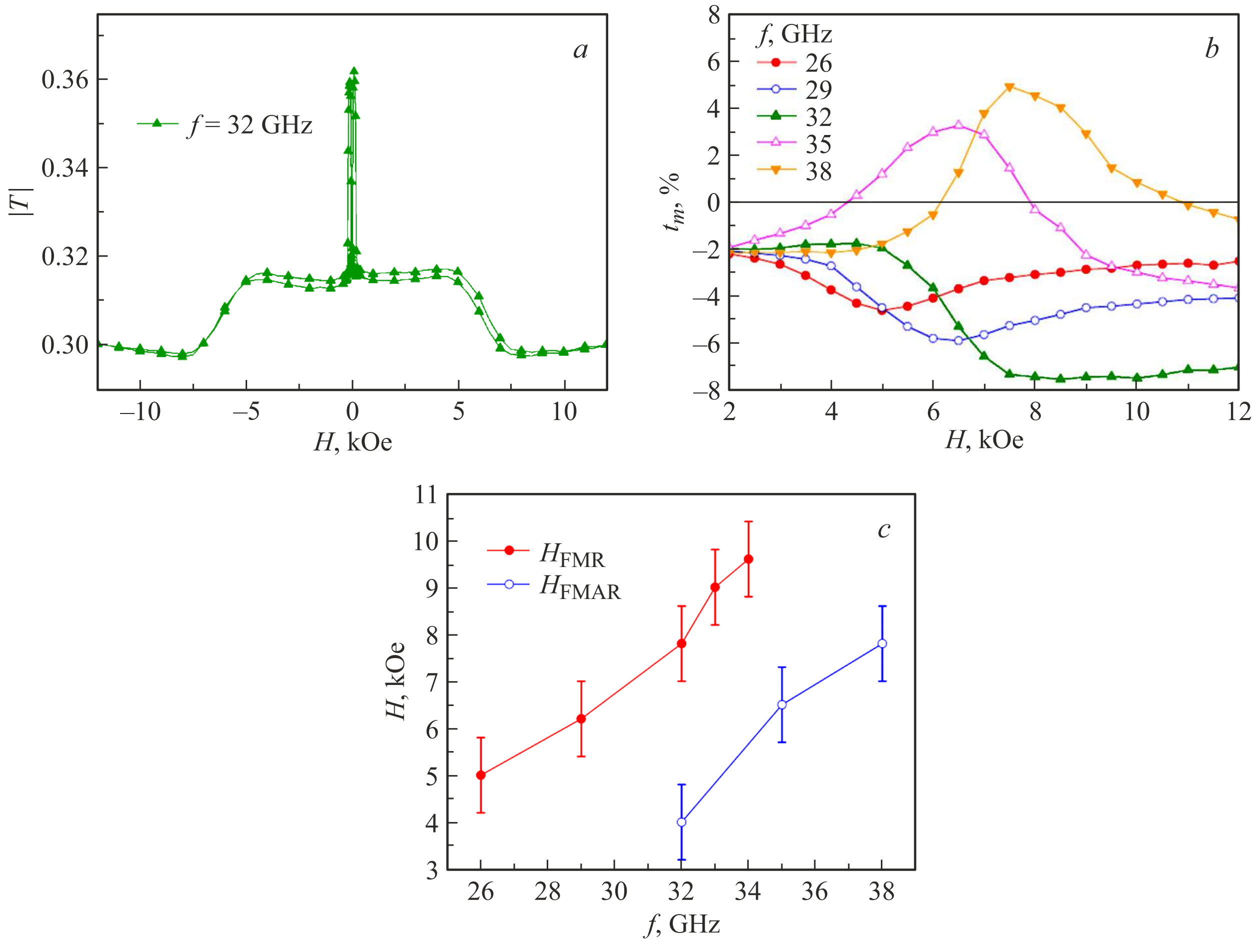

Figure 6. Magnetic field dependence of the transmittance modulus for a three-layer № 3 nanostructure sample: dependence measured at $f = 32$ GHz (*a*); dependences measured in the field range from 2 to 12 kOe at several frequencies (*b*); FMR and FMAR spectra constructed from extremums of transmittance and reflectance dependences (*c*).

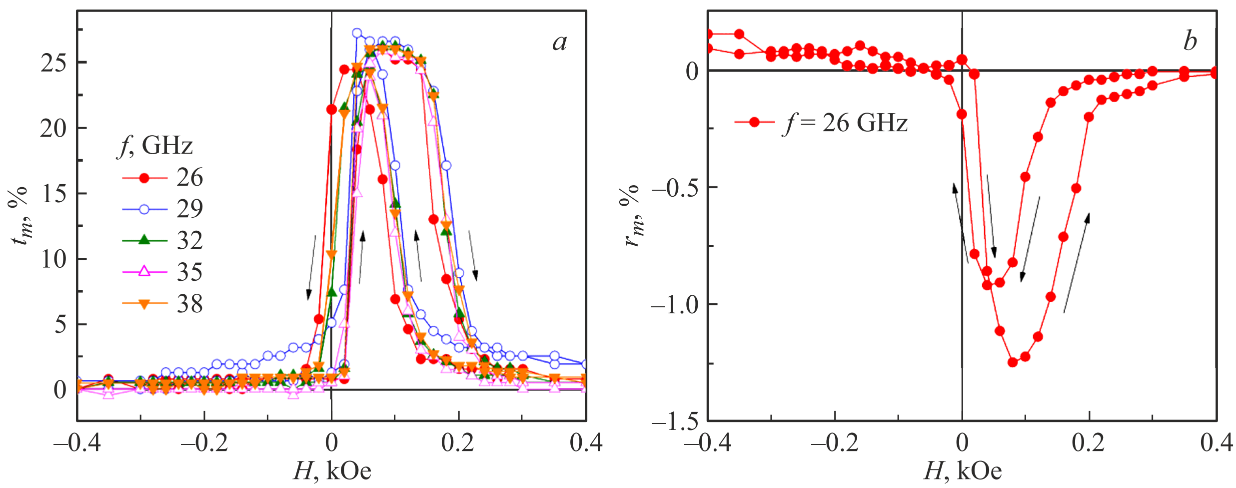

Figure 7. Dependences on the magnetic field of the microwave coefficients for the spin valve sample № 1: measurements of the transmission coefficient at several frequencies (*a*); measurements of the reflectance at $f = 26$ GHz (*b*).

relative magnetoresistance r:

$$t_m = r. \quad (2)$$

A one-to-one correspondence (2) is fulfilled for Fe/Cr, Co/Cu, AgPt/Co [26–28] super-

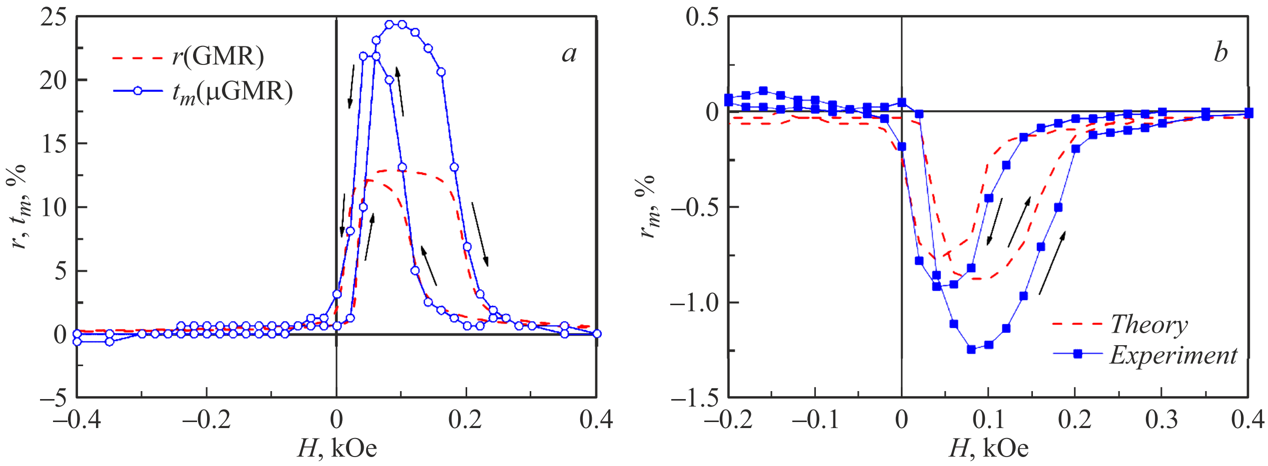

Figure 8. Comparison of magnetoresistance and transmittance variations at $f = 26$ GHz (*a*); experimentally measured at 26 GHz and calculated from transmittance measurements using the formula (3) (*b*).

lattices. In the [17] work, where spin valves Ta(10)/NiFe(3)/IrMn(6)/CoFe(1.5–3)/Cu(2.5)/CoFe(1)/NiFe(2)/Ta(2), it is concluded that the relative change in the transmittance must be equal to twice the relative magnetoresistance. The difference with (2) is due to the fact that in [17] the microwave transmittance was introduced as the ratio of the transmitted power to the incident power, in contrast to our definition of this value as the ratio of the transmitted wave amplitude to the incident power. If $t_m \ll 1$ and $r \ll 1$, then formula (1) and the conclusions drawn in [17] are equivalent. However, in [16], it has been observed that the relative changes in the microwave transmission coefficient exceed r. This was observed both for superlattices (CoFe/Cu)n and for CoFe/Cu/CoFe three-layer nanostructures. For three-layer nanostructures $t_m > r$ even at $r \approx 0.1 \ll 1$. In [18], the deviation from equality (2) is associated with the accepted assumption of replacing the multilayer nanostructure by a homogeneous film with effective parameters. This approximation does not take into account interlayer boundaries and the conditions of fixation of spins on them. Therefore, formula (1) should be treated as an approximation. Figure 8,*a* compares the magnetic field dependence of the magnetoresistance r measured at direct current and the relative changes in the transmittance at 26 GHz in the spin valve sample № 1. You can see that when the type of dependence is similar, the amount of change t_m is much larger r. According to equality (2), the value t_m is independent of frequency if there is no frequency dispersion of conductivity. The data in Fig. 5,*a* for the three-layer nanostructures and Fig. 7,*a* for the spin flaps suggest a weak frequency dependence of the microwave changes.

Now let us analyze the field dependence of the reflectance. In the previously used approximation of effective parameters, the field dependences of the transmission and reflectances are related by the relation [23]:

$$r_m = -T(0)(1 - T(0))t_m, \quad (3)$$

where

$$T(0) = \frac{2\rho}{Zd\mu(0)}.$$

Here ρ — effective conductivity of the nanostructure at $H = 0$, d — total thickness of metal layers of the nanostructure, $\mu(0)$ — magnetic permeability at $H = 0$. At millimeter-range frequencies in the materials in question, $\mu(0) \approx 1$. The impedance of a rectangular waveguide Z is calculated using the formula [25,29,30]:

$$Z = \sqrt{\frac{\mu_0\varepsilon_0}{1 - (\lambda/\lambda_c)^2}}, \quad (4)$$

where $\lambda = c/f$ — wavelength in free space, c — speed of propagation of electromagnetic wave in vacuum, $\lambda_c = 2a$ — critical wave length of waveguide, $a = 7.2$ mm — the larger cross-sectional dimension of the shielded waveguide of rectangular cross-section, $\mu_)$ and ε_0 — the magnetic and dielectric permittivities of the vacuum. For the spin valve № 1 $d = 39$ nm, $\rho = 0.483 \cdot 10^{-6}\,\Omega\cdot$m [14], $Z = 612\,\Omega$ at frequency 26 GHz. A comparison of the experimentally measured dependence of the microwave reflectance and the dependence calculated by formula (3) from the dependence of changes in the transmittance coefficient t_m is given in Fig. 8,*b*. We can see the similarity of the type of these dependencies, the approximate correspondence of the fields of extremums, but there is a discrepancy in the magnitude of changes. In particularly, the experiment gives for the reflectance at 26 GHz the maximum change in −1.3%, and the calculation — the value $r_m \approx -0.7\%$. As above, we attribute this difference to the influence of interlayer boundaries and to the fixation of spins on the boundaries. The formula (3), in the derivation of which the effective medium approximation was used, should also be considered an approximation.

Conclusion

A study of the microwave properties and magnetoresistance of spin valves and three-layer nanostructures has been performed. CoFe/Cu/CoFe/FeMn spin valves with high magnetoresistance and small hysteresis in the area of weak fields were chosen for the study. For comparison, measurements were made on three-layer CoFe/Cu/CoFe nanostructures with no FeMn antiferromagnetic layer. Spin valves and three-layer nanostructures were grown by magnetron sputtering.

It was found that the dependences of the microwave transmission coefficient on the magnetic field are caused by the microwave giant magneto-resistive effect. They are similar in shape to the magneto-resistance measured at direct current. It has been reliably proven that in CoFe/Cu/CoFe/FeMn spin valves, the μGMR-induced microwave changes far exceed the relative magnetoresistance in magnitude. The difference is attributed to the influence of interfaces in the nanostructures and to the spin fixation on the interfaces. In the considered frequency interval of 26–38 GHz, there is practically no frequency dependence of changes in the microwave transmittance coefficient. In the results of this study, we can conclude, that CoFe/Cu/CoFe/FeMn spin valves retain magneto-resistive properties at frequencies at least up to 38 GHz. The changes in microwave magneto-resistance for CoFe/Cu/CoFe three-layer nanostructures are symmetrical with respect to the $H = 0$ line, while for CoFe/Cu/CoFe/FeMn — spin valves are sharply asymmetrical. Similar is observed for the magnetoresistance dependences. Changes in the microwave reflectance are opposite in sign to changes in the transmittance coefficient.

Financial support of work

This work was performed within the topics „Spin" № 122021000036-3 and „Function" № 122021000035-6. Microwave measurements were carried out with support from the Russian Foundation for Basic Research, Grant № 20-02-00135.

Conflict of interest

The authors declare that they have no conflict of interest.

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