

Reduction of superparamagnetic clusters in the $[\text{Co}/\text{Cu}(111)]_n$ nanofilms, induced by the quantum size effect

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It is known that the quantum size effects are important for the formation of morphological properties of metal films. The regularities in the behavior of the superparamagnetic magnetoresistive effect in multilayer nanofilms $\text{Co}/\text{Cu}(111)$ in a magnetic field, found in the work, indicate the influence of the electron size effect on the formation of clusters in these films. The results of measurements of the high-field magnetoresistive effect are reported for multilayer films $[\text{Co}/\text{Cu}(111)]_{20}$ with a constant thickness of cobalt layers and the thickness of copper layer varying from film to film. It is found that an effective size of superparamagnetic formations is reduced in the films with thickness of the copper layers corresponding to the maxima of the antiferromagnetic exchange coupling between cobalt layers. It is suggested that the observed "grinding" of superparamagnetic particles is caused by oscillating changes in the electron density in the interface layer Co/Cu , induced by electron quantum size effect in the copper layers. © 2012 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4752100>]

1. Introduction

Layer nanostructures of the type of ferromagnet-normal metal (FM-NM) - multilayer films, pillars, wires, membranes, which are based on homogeneous or granular layers of Co and Cu - continue to be a primary focus of engineers and researchers of magnetoresistive materials.¹⁻³ The achieved value of the magnetoresistive effect at room temperature in nanostructures Co/Cu exceeds 90%.² The primary factor responsible for the magnetoresistive properties of layered and granular systems FM-NM is the presence of a sharp interface between the components. Nonwettability and mutual insolubility of Co and Cu prevents from the formation of alloys $\text{Co}_x\text{Cu}_{1-x}$ with intermediate magnetic properties⁴ that makes it possible to prepare sharp interfaces Co/Cu , resulting in the fact that systems based on them are among the most promising magnetoresistive materials.^{5,6}

A characteristic feature of multilayer nanofilms $[\text{Co}/\text{Cu}]_n$ prepared by different ways is the presence of superparamagnetic (SPM) clusters containing a large number of cobalt ions.⁷⁻¹² The presence of clusters is especially typical for films containing layers $\text{Cu}(111)$. The resistance of layered structures $[\text{Co}/\text{Cu}(111)]$ changes in a magnetic field similar to the change of resistance of granular Co in the matrix of Cu,^{13,14} Ag,¹⁵ Au.¹⁶ Even in high-quality films prepared by molecular epitaxy in a high vacuum, the main contribution to the magnetoresistance effect is the scattering of electrons on SPM clusters rather than on the antiferromagnetic exchange-coupled layers of Co.¹² The latter contribution is often only a few percent of the total magnetoresistive effect.^{10,12} This fact has even prevented an experimental verification of the existence of the exchange interaction between the Co layers in the $\text{Co}/\text{Cu}(111)/\text{Co}$ structures.^{17,18} The

investigation of causes of magnetic inhomogeneities in multilayered films based on ferromagnetic and noble metals and study of possibilities to control the processes of cluster formation have been carried out in many papers.

In this paper, attention is paid to the experimentally identified characteristics of magnetoresistive properties of multilayer nanofilms $[\text{Co}/\text{Cu}(111)]_{20}$ with varying thickness of copper layers. These features are that the size of SPM clusters, where the values of the magnetic moments were determined by fitting of experimental dependences of high-field magnetoresistance on a magnetic field to Langevin functions, depends nonmonotonically on the thickness of copper layers and at some values decreases significantly. The observed regularity points to the influence of the electron quantum size effect in copper layers on the formation of SPM clusters of cobalt atoms in the layered structure $\text{Co}/\text{Cu}(111)$.

2. Samples and experimental methods

The multilayer films $[\text{Co}/\text{Cu}]_{20}$ were deposited on mica substrates by magnetron sputtering. The substrate temperature during deposition of layers were close to 300 K. The residual atmosphere in the vacuum assembly was 10^{-6} Torr and working pressure of argon during the sputtering $1.3 \cdot 10^{-3}$ Torr. The deposition rate for Co and Cu were equal to 0.045 and 0.058 nm/s respectively. To ensure uniform growth of layers, first, the 5 nm-thick copper layer was deposited on mica. On top of it, by sequential condensation, layers of Co and Cu were formed. The thickness of the layers was determined by the regime and the time of deposition, and controlled by methods of multi-beam optical interferometry and small-angle X-ray diffraction. The error in determination of the layer thickness was less than 3%.

An effective thickness of the cobalt layers in all the films was identical and close to 0.8 nm. An effective thickness of the copper layers, d_{Cu} , for each film was constant and in different films it was 0.7, 0.8, 0.9, 1, 1.1, 1.2, 1.35, 1.5, 1.7, 1.8, 1.9, and 2 nm. Top layers of $[Co/Cu]_{19}/Co$ were covered with the copper layer of 1.25 nm. Layers of metals grew on each other, forming multilayer grains-pillars $Cu(1.25\text{ nm})[Co(0.8\text{ nm})/Cu(d_{Cu})]_{19}/Co(0.8\text{ nm})/Cu(5\text{ nm})$ with single-crystalline layers of fcc Co and Cu. The crystallographic planes (111) of layers were parallel to a substrate. Electron-microscopy investigations revealed that grains-pillars had transverse dimensions of about 8–10 nm. Despite the fact that the crystallographic “easy” axis [111] of the anisotropy of Co films is perpendicular to a substrate, magnetic moments are oriented under the influence of the demagnetization field in the plane of the layers. The magnetization curves of the films were recorded by means of magneto-optical methods and with the help of SQUID-magnetometry. The magnetoresistance of the films was measured at room temperature by using a standard four-contact method. The measurements geometry was longitudinal: a magnetic field was applied in the film plane parallel to an electric current. In addition, the SQUID-magnetometer was used for thermomagnetic measurements of one of the films.

3. Experimental results and discussion

Earlier studies of the longitudinal magneto-optical Kerr effect in these films has showed that the coercive fields of hysteresis magnetization loops in the field lying in the film plane has not exceeded 100 Oe for all films. We do not focus our attention on the magnetoresistance in such weak fields, and consider the field dependences of the magnetoresistance $MR(H)$ in fields above the magnetic saturation field of ferromagnetic components of layers. The value of the magnetoresistive effect is defined here as $MR = 100(R(H) - R(H \rightarrow 0)) / R(H \rightarrow 0)$. The value $R(H \rightarrow 0)$ was obtained by extrapolating the dependences $R(H)$ from a few kilo-Oersteds to zero field. The dependences $MR(H)$ have a common form typical of granular and multilayer structures ferromagnet-normal metal with different composition (Co–Ag,¹⁵ Co–Cu,⁸ Co/Cu,^{9,13} Ni–Cu/Cu,¹⁴ Co/Ag (Ref. 19)). Field dependences of the magnetoresistance in the films Co/Cu(111) studied by us have a form far from being saturated at $H = 15$ kOe (the maximum field in the experiment), whereas the magnetization of ferromagnetic components is close to full saturation in fields of about 1 kOe. Fig. 1 shows the dependences $MR(H)$ obtained for some films. The dependences for the rest of the films studied are similar.

As known, the giant magnetoresistance of layered and granular structures FM-NM appears due to the spin-dependent scattering of conduction electrons.^{5,20,21} Its value is proportional to cosine of the angle θ_{ij} between magnetic moments of scattering ferromagnetic centers. Reducing the angle θ_{ij} upon the alignment of magnetic moments of FM layers and granules along magnetic field decreases the probability of scattering of conduction electrons with the spin polarized along the direction of the magnetic field and thus reduces the resistance. In the films studied, the magnetization of the ferromagnetic layers has almost no effect on the dependences $MR(H)$. Almost all of the changes are due to

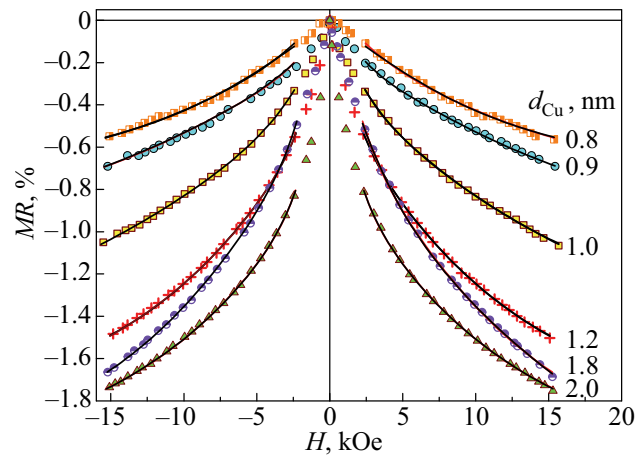


FIG. 1. Dependences of magnetoresistance for a few films in the series $[Co(0.8\text{ nm})/Cu(d_{Cu})]_{20}$ on a magnetic field, obtained in the longitudinal geometry of the experiment. The lines which superimposed each other in pairs show the fit of the experimental points by the expressions (1) and (2), starting with the field 2 kOe.

scattering of electrons on magnetic inhomogeneities, the magnetization of which remains far from saturation in a field of 15 kOe. Note that the magnetic inhomogeneities related to the existence in ferromagnetic films of steps, ledges, bridges between ferromagnetic layers are saturated in fields slightly higher than the saturation field of the ferromagnetic layers. The initial parts of the curves $MR(H)$ for films with thickness of copper layers 0.9 and 1.8 nm, in which one can expect maxima of the antiferromagnetic exchange interaction between cobalt layers, reflect, perhaps, the destruction of the antiferromagnetic order in the layers. The linear changes of the magnetization in a magnetic field, arising in the process of collapsing the magnetic moments of the Co layers, are only clearly visible in the film $[Co/Cu(1.8\text{ nm})]_{20}$ in the fields up to 2 kOe. Therefore, we will only consider changes in the magnetoresistance in fields higher than 2 kOe, where almost all of the changes are due to magnetization of SPM particles.

Superparamagnetic formations in the films can be both SPM clusters formed in a copper layer during the deposition of the film, and the parts of cobalt layers, weakly coupled with the parent ferromagnetic mass (“superspin flakes”), or completely detached from it (“superspin peels” – “loose spins”) appeared near the interface.⁸ Expected dependences of the magnetoresistance for these SPM structures may be slightly different. Thus, for isolated SPM particles located far from the ferromagnetic layer, the $MR(H)$ should change similar to the granular SPM systems. Namely, it is determined by an average value $\langle \cos \theta_{ij} \rangle$, where θ_{ij} is the angle between the magnetic moments of any pair of adjacent SPM particles, located in the sphere with a radius equal to the average length of electron free path. If the interaction of SPM particles with each other can be neglected, their magnetic moments are aligned along an external magnetic field, independently of each other, and $\langle \cos \theta_{ij} \rangle = \langle \cos \varphi_i \cos \varphi_j \rangle$, where φ_i and φ_j are angles between the magnetic moments of each of the SPM particles and the external magnetic field. Since $\langle \cos \varphi \rangle = M(H)/M_S = L(\mu H/k_B T)$, where $L(\mu H/k_B T)$ is the Langevin function, k_B is the Boltzmann constant, then for the dependences $MR(H)$ of the SPM particles with the same size the relation $MR(H) = AL^2(\mu H/k_B T)$ is true.²² In the presence

of SPM particles of different sizes one can expect a more complex relationship,

$$MR(H) = [A_1L(\mu_1H/k_B T) + A_2L(\mu_2H/k_B T) + A_3L(\mu_3H/k_B T) + \dots]^2.$$

For SPM formations located in the immediate vicinity of ferromagnetic layers, “superspin peels”, the field dependence of the magnetoresistance is mainly caused by scattering of conduction electrons at the interface FM/NM and on boundaries of the SPM formations. In fields above the saturation field of the ferromagnetic layer, $\langle \cos \varphi \rangle = 1$. In this case, the magnetoresistive effect should be described by the Langevin function, or a sum of Langevin functions, assuming the presence of SPM particles with different sizes^{12,22}

$$MR(H) = B_1L(\mu_1H/k_B T) + B_2L(\mu_2H/k_B T) + B_3L(\mu_3H/k_B T) + \dots$$

Because the Co and Cu layers in the films have a thickness of only a few atomic layers, a superposition of all the dependences is expected.

We tried to describe the observed dependences within these simple models. For simplicity, we restrict ourselves to the assumption that the system has superparamagnetic particles of one or two sizes. Fig. 2 shows the fits of experimental dependence $MR(H)$ of the film with $d_{Cu} = 1.8$ nm to the

Langevin functions. It is seen that the experimental data cannot be described by a single Langevin function (Fig. 2(c)) or its square (Fig. 2(a)), but can be well enough described by a superposition of Langevin functions within both models ((Figs. 2(b) and 2(d)). It is difficult to chose one of them as the best fit. The magnetic moments of the SPM formations in all the films were determined as adjustable parameters for the best fit of experimental data to the two dependences

$$MR(H) = [A_1L(\mu_1H/k_B T) + A_2L(\mu_2H/k_B T)]^2 \quad (1)$$

and

$$MR(H) = B_1L(\mu_1H/k_B T) + B_2L(\mu_2H/k_B T). \quad (2)$$

As seen in Fig. 1, the experimental data are described equally well by Eqs. (1) and (2). Fig. 3(a) presents the harmonic means of the magnetic moments for the SPM particles in the films as a function of thickness of the copper layer, manifested themselves in the magnetoresistive effect. The harmonic means were determined by the expressions

$$\langle \mu \rangle_{bq} = \frac{A_1 + A_2}{A_1/\mu_1 + A_2/\mu_2} \quad (3)$$

for biquadratic and

$$\langle \mu \rangle_{bl} = \frac{B_1^2 + B_2^2 + 4B_1B_2}{(B_1^2 + 2B_1B_2)/\mu_1 + (B_2^2 + 2B_1B_2)/\mu_2} \quad (4)$$

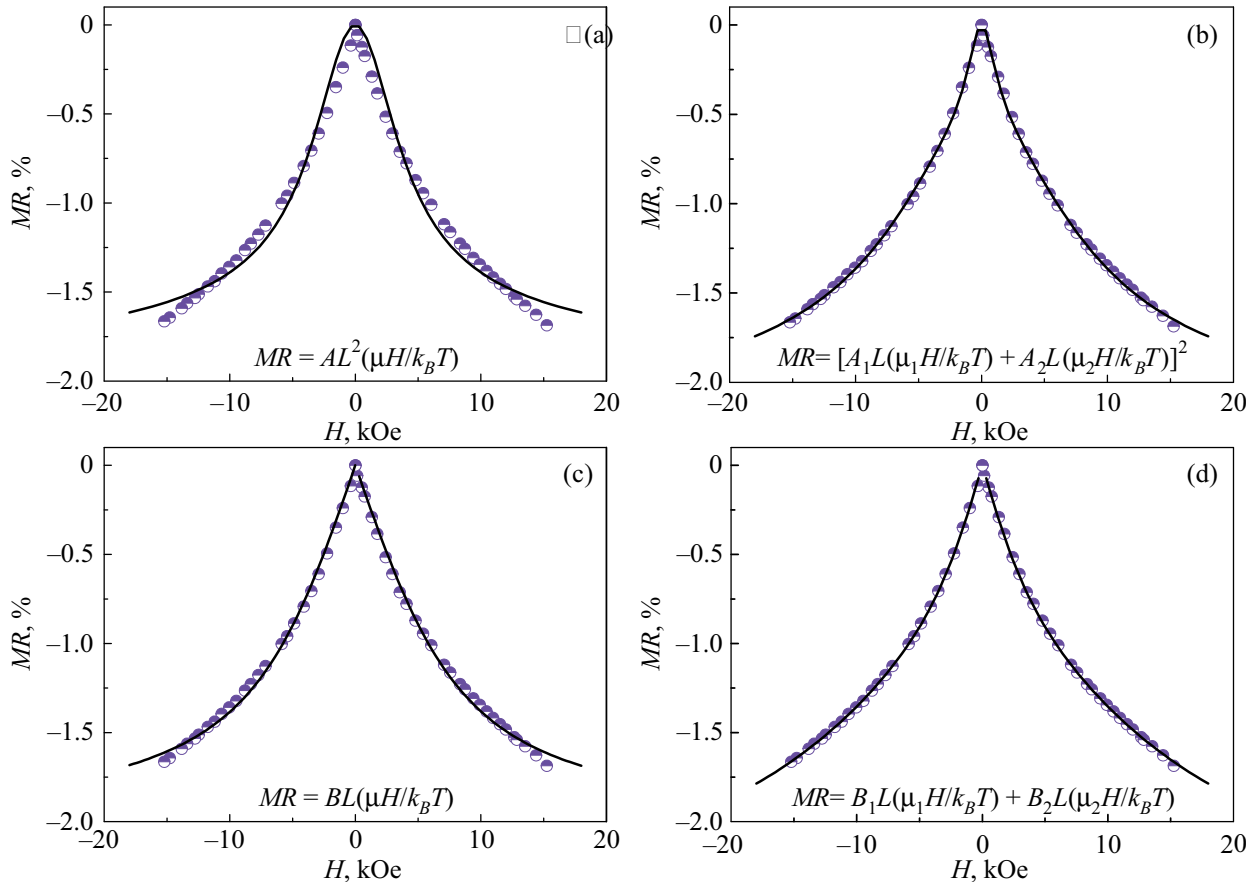


FIG. 2. Fits of an experimental dependence of magnetoresistance on a magnetic field $MR(H)$ of the film $[\text{Co}(0.8 \text{ nm})/\text{Cu}(1.8 \text{ nm})]_{20}$ to: a square of the Langevin function (a), a square sum of two Langevin functions (b), the Langevin function of the first order (c), a sum of two Langevin functions (d).

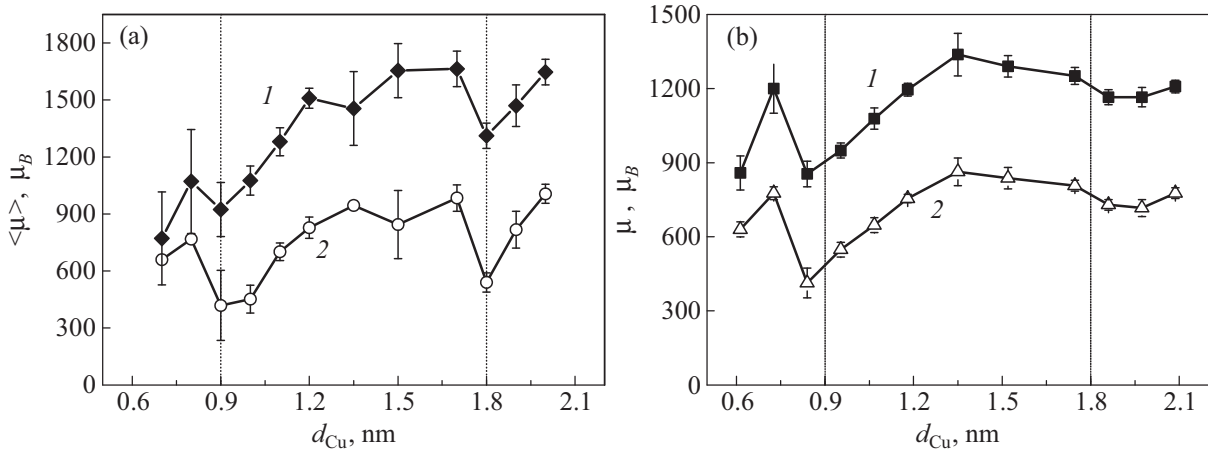


FIG. 3. Harmonic weighted mean values of magnetic moments of the SPM particles depending on the thickness of a copper layer in the multilayer films $[Co(0.8 nm)/Cu(dCu)]_{20}$, obtained from fitting the experimental dependences $MR(H)$, starting with the field 2 kOe, to a square sum (curve 1) and a sum of two Langevin functions (curve 2) (a). Magnetic moments of the SPM particles obtained from fitting experimental data, starting with the field 5 kOe, to a square of one Langevin function (curve 1) and one Langevin function (curve 2) (b).

for bilinear approximation. It can be seen in Fig. 3(a) that the changes of magnetic moments when varying the thickness of copper layers have the particular regularities: in the films with $d_{Cu} = 0.9 nm$ and $d_{Cu} = 1.8 nm$, the reductions of their magnetic moments are well evident. The values of the SPM moments for all films, determined by fitting the experimental data for fields above 5 kOe to one Langevin function and its square, are shown in Fig. 3(b). A decrease of the SPM moments for the same films can also be seen there.

The different behavior of the magnetoresistance effect in $[Co(0.8 nm)/Cu(0.9 nm)]_{20}$ and $[Co(0.8 nm)/Cu(1.8 nm)]_{20}$ is also showed up in the dependences of the specific contributions to the magnetoresistance effect, accounted for a single magnetic moment of the SPM particle at saturation, A_i/μ_i (Fig. 4). The values of the magnetic moments of clusters μ_i and their contributions to the effect of A_i were obtained by fitting the experimental data to a sum of two Langevin functions. Bursts in the specific effect are, apparently, due to an increase in the total surface interface of SPM Co/Cu in these films and an increase in the efficiency of clusters consisting of several hundred cobalt atoms.²³ For the films with $d_{Cu} = 0.9 nm$ and $d_{Cu} = 1.8 nm$ a number of cobalt ions in the harmonically averaged clusters $\langle \mu_i \rangle / 1.67 \mu_B$ is about 250 and 320 respectively.

Note that if the SPM particles interact with each other, the magnetic moments defined in this way are effective. Their true values μ_{true} can be higher for an antiferromagnetic interaction and lower for ferromagnetic one.^{24,25} In order to obtain an information about the type of interaction between the SPM particles, we measured the temperature dependences of the magnetic moment of the film $[Co/Cu(1.8 nm)]_{20}$ in a field of 50 Oe during warming after cooling without field and in a field of 700 Oe. Fig. 5(a) shows the corresponding curves $M_{FC}(H)$ and $M_{ZFC}(H)$. Both curves have a form typical of a system of superparamagnetic clusters with a blocking temperature of their main part of about 100 K. The curves ZFC/FC bifurcate at the temperature close to 120 K. The difference between the temperature of maximum in the ZFC dependence ($T_{max} = 100 K$) and the temperature of bifurcation of the curves indicates the existence of interaction between the SPM clusters. The inset shows the

dependence of the inverse susceptibility on temperature, where it is seen that the character of the interaction between large clusters, giving a major contribution to the magnetic susceptibility at high temperatures, is ferromagnetic with a characteristic interaction temperature Θ close to 225 K. However, the ferromagnetic interaction between smaller clusters, as evident from the low-temperature tail of the dependence $H/M(T)$, is much smaller and becomes antiferromagnetic. Following Ref. 24, one can estimate the “true” cluster dimensions which manifest themselves in the magnetoresistive properties of the films with $d_{Cu} = 1.8 nm$. A greater contribution to the magnetoresistive effect is given by smaller clusters, the interaction between which is antiferromagnetic. In this case, the size of the “true” clusters, μ_{true} , is slightly bigger than that determined from fitting the experimental data to the Langevin dependences μ ,

$$\mu_{true} = \frac{\mu(T + T^*)}{T}. \quad (5)$$

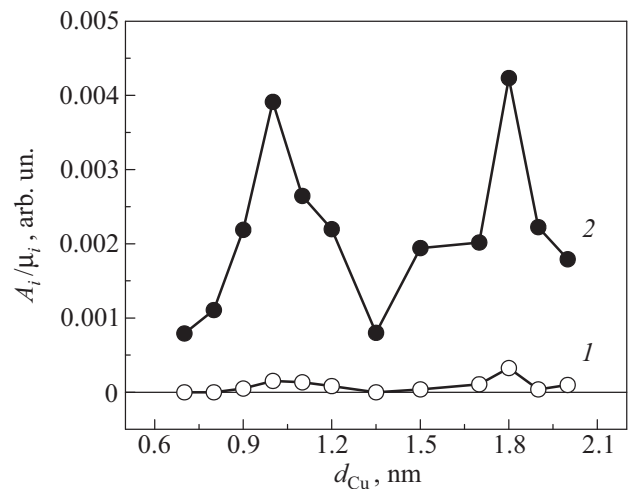


FIG. 4. Dependences of specific contributions to the magnetoresistive effect for two groups of the SPM particles A_i/μ_i on the thickness of the copper layer of the films $[Co(0.8 nm)/Cu(dCu)]_{20}$, obtained by fitting experimental data to a sum of two Langevin functions. The lines (1) and (2) correspond to the SPM particles of larger and smaller size respectively.

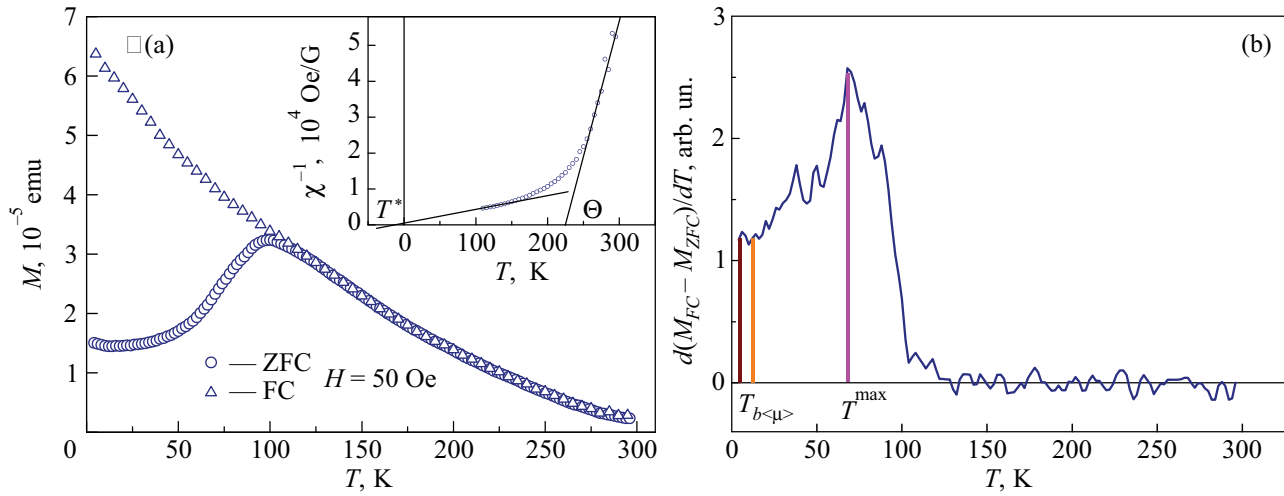


FIG. 5. Temperature dependences of a magnetic moment of the film $[\text{Co}/\text{Cu}(1.8 \text{ nm})]_{20}/\text{mica}$, obtained on warming the sample in a field of 50 Oe after prior cooling without a field ($M_{ZFC}(H)$) and in the field $H = 700 \text{ Oe}$ ($M_{FC}(H)$) (a). $d(M_{FC} - M_{ZFC})/dT$ is the distribution of blocking temperatures of clusters with different size (b). Vertical lines indicate the blocking temperatures of "fine" ($540 \mu_B$, $T_b = 5 \text{ K}$) and "large" ($1353 \mu_B$, $T_b = 12.4 \text{ K}$) clusters, magnetic moments of which were obtained as harmonic means from fitting the experimental magnetoresistive dependences $MR(H)$ by a sum and a square sum of the Langevin functions respectively. The distribution maximum at T^{max} corresponds to clusters with the magnetic moment $8500 \mu_B$.

However, since $T^* \lesssim 20 \text{ K} \ll T = 295 \text{ K}$, the difference between μ_{true} and μ is insignificant.

In Fig. 5(b) the derivative $d(M_{FC} - M_{ZFC})/dT$ shows the distribution of blocking temperatures of the SPM clusters. Neglecting the weak coupling between small clusters, which give the main contribution to the magnetoresistive effect, and assuming that with decreasing the temperature their magnetic moments are blocked by magnetic anisotropy, one can determine the blocking temperature $T_b = KV_{cl}/25k_B$ of clusters in the film $[\text{Co}/\text{Cu}(1.8 \text{ nm})]_{20}$, which showed themselves in the magnetoresistive effect. Here, K is the magnetic anisotropy constant, V_{cl} is the cluster volume. To calculate T_b we used the anisotropy constant $K = 4.5 \cdot 10^5 \text{ J/m}^3$ of bulk Co (fcc).²⁶ The volume of one cluster $V_{cl} = V_0 \mu_{MR}/\mu_0$ was calculated by using the value of the magnetic moment of the Co ion ($\mu_0 = 1.67 \mu_B$) and the volume occupied by one ion of cobalt in the fcc structure is $V_0 = 11.76 \cdot 10^{-24} \text{ cm}^3$. In Fig. 5(b) the vertical lines indicate the blocking temperatures $T_{b(\mu)} = 5$ and 12.4 K of clusters with the magnetic moments $\langle \mu \rangle = 540 \mu_B$ and $1350 \mu_B$, which were defined as harmonic means obtained from fitting the curves $MR(H)$ by sum and square sum of the Langevin functions respectively. The distribution maximum T^{max} is assignable to clusters with the magnetic moment $8500 \mu_B$, and the "magnetoresistive" clusters are on the low-temperature "tail" of the distribution.

The thickness of copper layer $d_{\text{Cu}} = 0.9 \text{ nm}$ and $d_{\text{Cu}} = 1.8 \text{ nm}$, for which the decreasing of dimensions of the SPM clusters was found, corresponds to the maxima of AFM exchange coupling between layers of Co via Cu.²⁷ In the confined Cu layers of this thickness there occurs a quantum size effect, at which the energy of the resonant states of conduction electrons coincides with the Fermi level of Cu.²⁸ This leads to a redistribution of electron density at the interface, which may significantly change the interaction of Co atoms with atoms of Cu. It is known that the resonant electronic effects may lead to changes in the structure of the atomic lattice of a metal layer,²⁹⁻³¹ they often affect significantly the growth scenario of metal and semiconductor nanofilms, responsible for self-organization of ensembles of

quantum dots,^{32,33} affect the structure of magnetic clusters on the surface of a normal metal.³⁴

The found reduction in the size of the SPM particles in the investigated films $[\text{Co}(0.8 \text{ nm})/\text{Cu}]_{20}$ is, apparently, due to an influence of the quantum size effect on the appearance (during formation of an interface layer of Co/Cu) of single- or bilayer flat cobalt clusters, separated from the central area of the ferromagnetic cobalt layer by a diluted paramagnetic layer Co-Cu.

4. Conclusions

The performed studies of superparamagnetic magnetoresistive effect in multilayer nanofilms $[\text{Co}(0.8 \text{ nm})/\text{Cu}]_{20}$, obtained by magnetron sputtering, have revealed that the size of superparamagnetic structures forming in them depends nonmonotonically on the thickness of copper layers. For the thickness of copper layers, at which antiferromagnetic coupling between the ferromagnetic layers of cobalt appears, the size of superparamagnetic particles is much smaller than in films with other thickness of copper layers. It is suggested that the formation of much smaller superparamagnetic blocks is a manifestation of the electronic quantum size effect, when the formation in copper layers of the resonant electronic states leads to a change in the density of conduction electrons on the interface, modifies the conditions of accommodation of lattice structures for adjacent layers of cobalt and copper, and favors the formation of small isolated monoatomic areas in Co layers of the type of "superparamagnetic peel".

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