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Conductivity and magnetoconductivity of $CoFeB/SiO_2$ composite films with different structures at temperatures $2-360$ K and magnetic fields up to 9 T

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This paper studies temperature dependences of the conductivity and magnetoconductivity of $CoFeB/SiO_2$ composite films on a polymer substrate with different total concentrations of metals Co and Fe ($x = 41-74$ at.%) in the temperature range of $2-360$ K and in magnetic fields up to 9 T. It has been found that at low concentrations of $x = 41$, 47 at.%, the conductivity and magnetoconductivity of composite films increase with temperature in magnetic fields of 1 and 5 T. At high $x = 64$ and 73 at.%, the conductivity and magnetoconductivity of composite films are weakly dependent on temperature in the range $2-240$ K. It is shown that $CoFeB/SiO_2$ composite films with a minimum concentration of metals $x = 41$ at.%, with a granular structure and a small volume of percolation regions, have maximum negative and positive magnetoconductivity. It is shown that the temperature behavior of the conductivity and magnetoconductivity of composite films with different metal concentrations x is largely determined by the volume ratio of granular and metallic regions.

Keywords: composite metal-dielectric films, conductivity, magnetoconductivity, temperature dependence, magnetic field, structure.

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Composite materials consisting of metallic and dielectric nanoscale granules and micro-regions are of considerable scientific and practical interest [1–9]. $CoFeB/SiO_2$ composite films have unique magnetic, transport and microwave reflective properties, which makes them promising materials for applications in spintronics, magnetic sensors and other fields of nanoelectronics [1–4]. Charge transport in granular composite films is mainly governed by tunneling. It strongly depends on metal concentration, grain size, intergrain distance, temperature, and external electric and magnetic fields [5,6]. Studies of the magnetic properties of $CoFeB/SiO_2$ films with a granular-percolation structure [1] show that the structure of composite films has a significant role in their temperature behavior of magnetization and coercive force, which makes the study of the influence of the film structure on other physical properties, including conductivity and magnetoconductivity an urgent task of modern condensed matter physics and materials science. The aim of this work was to experimentally study the effect of metal concentration and the structure of $CoFeB/SiO_2$ composite films on the temperature behavior of conductivity and magnetoconductivity.

$CoFeB/SiO_2$ composite films were synthesized at Voronezh State Technical University (VSTU) on a vacuum unit UVN-2M [10]. The morphology and structure of composites and composite films of various compositions are described in detail in the doctoral dissertation of A.V. Sitnikov [10]. The ion beam sputtering method was used to obtain $CoFeB/SiO_2$ composite films. The film sputtering process was carried out in a working argon gas environment at a pressure of 26.7 mPa. Plates made of an alloy of $Co_{41}Fe_{39}B_{20}$ and silicon dioxide SiO_2 were used as sputtering targets. Ion beams from two sources simultaneously acted on metallic and dielectric targets. A polymer sheet made of polyethylene terephthalate (dacron) with dimensions $210-297$ mm and a thickness of $30\,\mu$m was used as a substrate for sputtering composite films. The target plates made of a metal alloy and a dielectric were positioned in such a way that the concentration of x CoFe metals in the composite layer increased along the length of the dacron sheet. $CoFeB/SiO_2$ composite films were obtained by direct sputtering in this paper and in Ref. [1], while $CoFeB/SiO_2$ composite films studied in Ref. [3] were obtained by inclined sputtering. The $CoFeB/SiO_2$ films studied in Ref. [2] were obtained by direct sputtering, but in an atmosphere of argon and nitrogen, therefore, for such films, the granular structure extends to high concentrations of metals x. To ensure greater uniformity of the thickness and composition of the formed composite films, the substrate holder rotated at a speed of 1 revolution per 5 min, and the entire deposition of the composite layer on the polymer sheet lasted 100 min. To

Table 1. Thicknesses and compositions of $CoFeB/SiO_2$ films

Number of sample	d, nm	x	Co	Fe	B	O	Si
1	556	41	22	19	4	39	16
2	741	47	25	22	4	33	16
3	864	64	34	30	5	20	11
4	759	73	39	34	10	11	6

determine the thickness of the films, cross-sectional images of the composite films on polymer substrates were taken using a TESCAN MIRA3 LMN electron microscope using secondary(SE) and back-reflected (BSE) electron detectors, which made it possible to visualize the film/substrate interface. The elemental composition of the films was also determined using an electron microscope and an energy dispersive X-ray microanalysis device. Table 1 shows the elemental composition and total concentration of metal atoms Fe, Co in at.%, as well as the thickness of composite films.

Studies of the temperature and magneto-field dependences of the electrical resistance of films were conducted at the Center for Diagnostics of Functional Materials of the St. Petersburg State University Science Park using the PPMS-9 (Physical Property Measurement System) measuring complex with the EverCool-II (Quantum Design) cryostat. The temperature of the studied samples using a cryostat could be set in the range of 2–400 K, and the magnetic field could vary from 0 to 9 T. The electrical resistance of the samples was measured using a standard four-probe method. The magnetic field was applied perpendicular to the plane of the films. The obtained values of the electrical surface resistance were converted into conductivity G. Based on the obtained conductivity data, the relative magnetoconductivity was calculated using the formula

$$\frac{\Delta G}{G_0} = \frac{G_B - G_0}{G_0},$$

where G_0 and G_B are the sample conductivities in a zero magnetic field and in an induction field B, respectively.

The surface relief of the films was studied using an atomic force microscope (AFM) NTEGRA PRIMA (NT-MDT, Russia) using a cantilever and a magnetic probe MFM10 [1,2]. The silicon probe was coated with a 40 nm thick CoCr magnetic alloy. The oscillation frequency of the external force acting on the cantilever with a magnetic probe was selected in the range 47–90 kHz, close to the natural oscillation frequency of the cantilever. 2×2 mm film samples were cut to study the films using AFM. The obtained relief images displayed the height and dimensions of homogeneous and heterogeneous surface areas of the samples. Figure 1 shows images of the surface relief of composite films for four metal concentrations $x = 41$, 47, 64, 73 at.%. As can be seen from Figure 1, for all films with different x, there are surface areas with large and small heights that correspond to granular and percolation or metallic regions. Magneto-phase contrast images of $CoFeB/SiO_2$ composite films of similar compositions are given in Ref. [1]. As can be seen from Figure 1, the average sizes and areas of SM metal regions increase with increasing metal concentrations x. The estimation of the areas of metal regions in composite films at different concentrations of x yields the following results: $S_M[10^{-12}\,\mathrm{m}^2] = 2$ at $x = 41$ at.%, 3 at $x = 47$ at.%, 8 at $x = 64$ at.% and 7 $x = 73$ at.%.

Figures 2, 3 show the temperature and reverse temperature dependences of conductivity for composite films with four different metal concentrations x, which are characterized by different sizes of granular and metallic regions. As can be seen from Figures 2, 3, for composite films No. 1 and No. 2 with a granular structure and a small volume of metallic regions, the conductivity of G increases exponentially in different magnetic fields in the temperature range from 100 to 360 K. The conductivity in a zero magnetic field increases from $7\cdot10^{-9}$ to $1.7\cdot10^{-6}\,\Omega^{-1}$ for film No. 1 and from $2.2\cdot10^{-8}$ to $3.7\cdot10^{-6}\,\Omega^{-1}$ for film No. 2 as the temperature increases from 100 to 360 K (see Table 2). The growth rate of the conductivity of films No. 1 and № 2 significantly increases with increasing temperature in the high temperature range of 280–360 K. An increase in the conductivity of composite films with increasing temperature indicates the predominance of the mechanism of tunneling or thermally activated jumps between metal granules with a characteristic activation energy of E_a. The activation energy E_a, determined by extrapolating the functional dependence of conductivity on the reverse temperature [7], decreases with increasing metal concentration for the temperature range of 100–250 K (Figure 3). For example, the activation energy of E_a is 0.22 eV at $x = 41$ at.% and 0.12 eV at $x = 47$ at.%. The conductivity of a composite film № 3 ($x = 64$ at.%) with granular regions and long metal bands [1] slowly increases with increasing temperature in the temperature range of 20–200 K in different magnetic fields. A greater increase in conductivity is observed with increasing temperature in the high temperature range of 200–360 K, for the film No. 3. The conductivity of a composite film No. 4 ($x = 73$ at.%) with extended metallic regions weakly depends on temperature over a wide temperature range 20–360 K at different magnetic fields. In the low temperature range from 2 to 20 K, a region of significant increase in conductivity with increasing temperature is observed for the film No. 4. For films No. 3 and No. 4, which contain a large fraction of metallic regions, the contribution of these regions to conductivity decreases with increasing temperature. In this case, the contribution to the conductivity of the granular structure of composite films increases with increasing temperature. The balance of the two contributions to the conductivity from the granular and metallic regions of composite films leads to a weak temperature dependence of the conductivity of the films.

As can be seen from Figure 4, the magnetoconductivity of composite films No. 1 and No. 2 with a granular structure

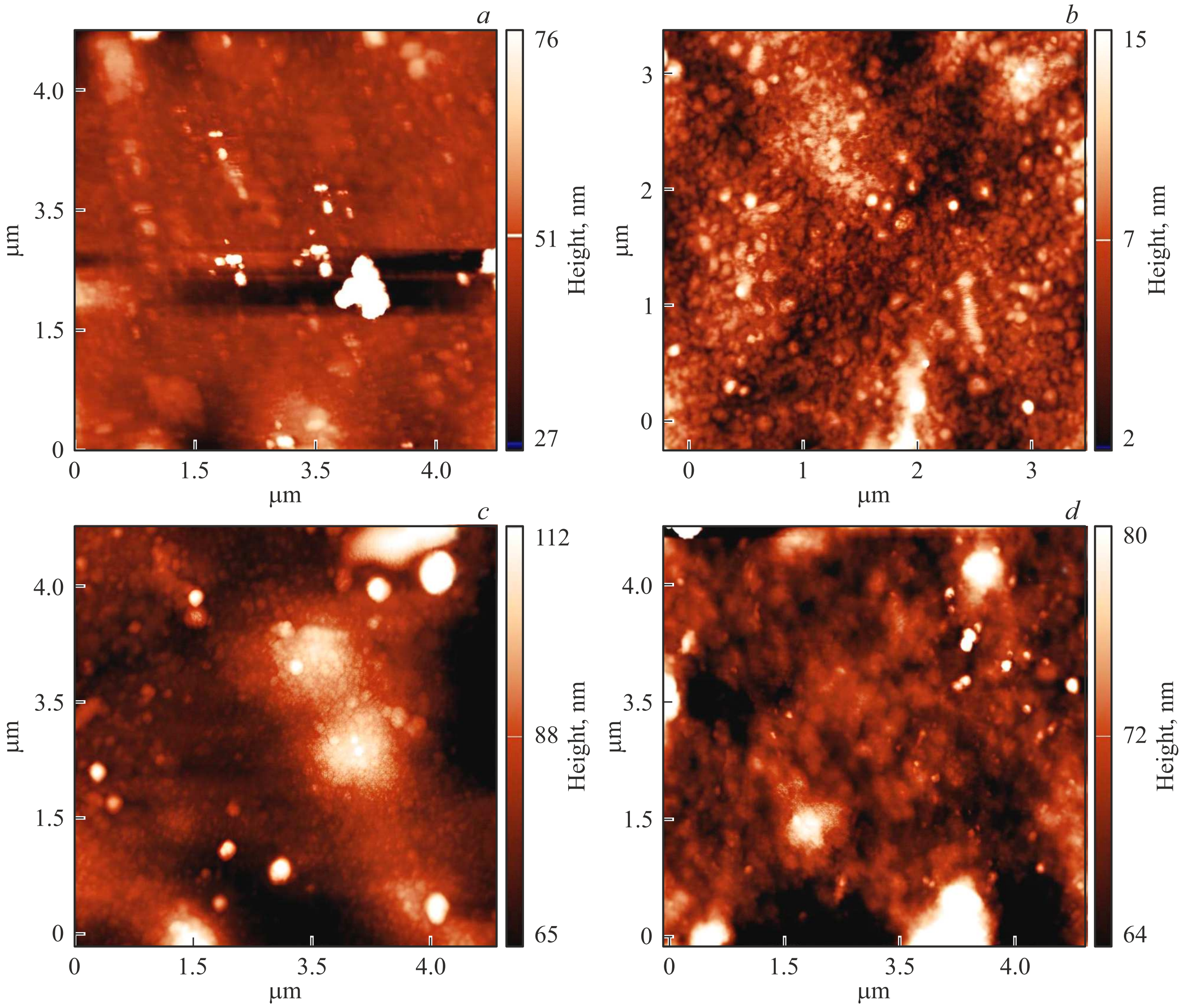

Figure 1. Images of the surface relief of $CoFeB/SiO_2$ composite films with a metal concentration of x, at.% = 41 (a), 47 (b), 64 (c), 73 (d). The values of the heights of the surface irregularities of the films in nanometers (nm) are shown on the right on the color scale.

Table 2. Conductivity of $CoFeB/SiO_2$ composite films at different temperatures and magnetic fields

T, K	Conductivity G									
	$G \cdot 10^9$, Ω^{-1}						$G \cdot 10^3$, Ω^{-1}			
2	0.3	0.1	0.1	4.1	4.1	3.1	3.3	3.2	72.7	72.9
10	0.4	0.1	0.1	4.7	4.7	3.6	3.4	3.2	73.2	73.1
50	0.9	0.4	0.4	8.9	9.2	7.3	3.4	3.3	73.8	73.8
100	6.9	1.4	1.3	19.6	21.2	17.8	3.4	3.3	74.1	74.1
150	7.9	5.3	4.5	43.4	49.0	43.3	3.4	3.3	74.2	74.2
200	15.1	20.0	16.1	96.2	112.9	105.5	3.5	3.3	74.2	74.2
250	117	75.4	57.2	213.0	260.2	256.9	3.5	3.4	74.1	74.1
300	275	283.6	203.9	471.7	599.6	625.5	3.7	3.5	73.9	73.9
350	786	1067	725.9	1044.5	1382.0	1523.3	3.9	3.7	73.7	73.6
B_0, T	0	1	5	0	1	5	0	9	0	9
Film No.	1	1	1	2	2	2	3	3	4	4

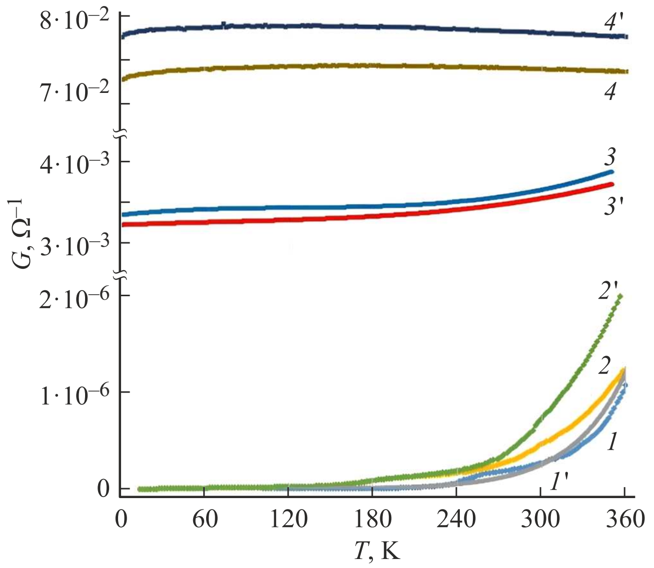

Figure 2. Temperature dependences of the conductivity of CoFeB/SiO_2 composite films with metal concentrations of $x = 41$ at.% (curves *1* and *1'*), 47 at.% (*2* and *2'*), 64 at.% (*3* and *3'*), 73 at.% (*4* and *4'*). The studies were carried out in magnetic fields with inductions $B = 0$ (curves *1, 2, 3, 4*), 5 T (*1', 2'*), 9 T (*3', 4 '*). The curve numbers and film numbers shown in Table 1 are the same.

and small percolation regions increases in magnetic fields with inductions 1 and 5 T over the entire temperature range studied. For a composite film No. 1, the magnetoconductivity increases from a negative value -0.62 to a positive value $+0.6$ with a magnetic field of 1 T and increases to 0 with a larger magnetic field of 5 T with an increase in temperature from 10 to 360 K. The magnetoconductivity of composite films No. 3 and No. 4 with a granular structure and a large volume of metallic regions in magnetic fields with inductions 1 and 5 T slightly decreases over the entire studied temperature range: from -0.02 to -0.07 for film No. 3 and from $+0.07$ to $+0.02$ for film *No.* 4 when the temperature increases from 10 to 360 K. The highest values of negative and positive relative magnetoconductivity, equal to -0.62 and $+0.6$ at temperatures of 2 and 360 K, respectively, are observed for a film No. 1 with a granular structure and with the smallest percolation regions in a 1 T magnetic field. Small values of positive and negative magnetoconductivity in a large magnetic field of 9 T, equal to $+0.07$ and -0.01 at temperatures of 2 and 360 K, are observed for a film No. 4 with a granular structure and extended metallic regions. The different behavior of the temperature dependence of the magnetoconductivity of CoFeB/SiO_2 composite films with different metal concentrations x can be explained by the competition of several conduction mechanisms: spin-dependent tunneling, which usually contributes to positive magnetoconductivity, and the classical Lorentz force effect, which contributes to negative magnetoconductivity. The temperature behavior of magnetoconductivity is also determined by other contributions to magnetoconductivity, such as the temperature dependence of the scattering intensity of electric charge carriers at the boundaries of granules and the low-temperature blocking effect of magnetic moments of granules [5,6]. The relative contribution of these mechanisms to magnetoconductivity will depend on the ratio of the volumes of the granular

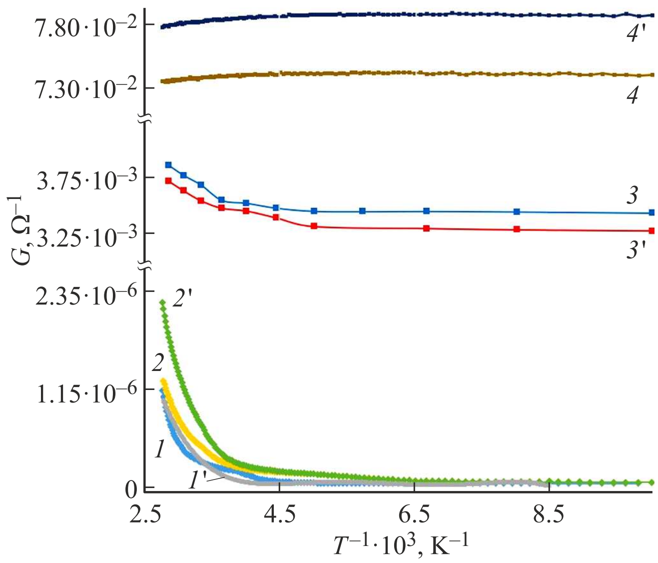

Figure 3. Reverse temperature dependences of conductivity for CoFeB/SiO_2 composite films with metal concentrations of $x = 41$ at.% (curves *1* and *1'*), 47 at.% (*2* and *2'*), 64 at.% (*3* and *3'*), 73 at.% (*4* and *4'*). The studies were carried out in magnetic fields with inductions $B = 0$ (curves *1, 2, 3, 4*), 5 T (*1', 2'*), 9 T (*3', 4 '*). The curve numbers and film numbers shown in Table 1 are the same.

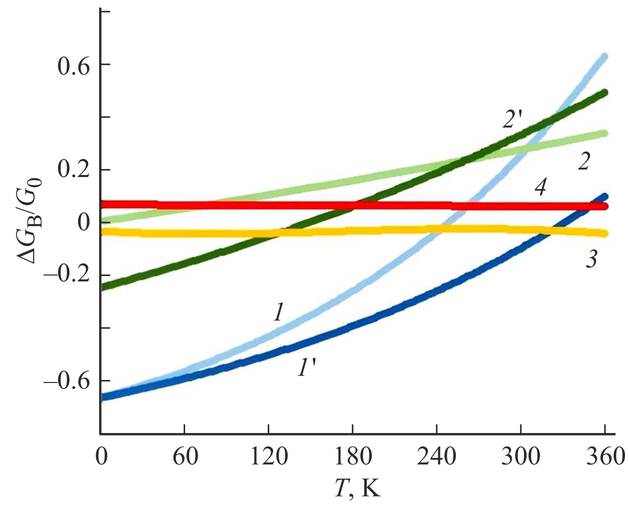

Figure 4. Temperature dependences of the relative magnetoconductivity of CoFeB/SiO_2 composite films with metal concentrations of $x = 41$ at.% (curves *1* and *1'*), 47 at.% (*2* and *2'*), 64 at.% (*3*), 73 at.% (*4*). The studies were carried out in magnetic fields with inductions $B = 1$ T (curves *1, 2*), 5 T (*1', 2 '*), 9 T (*3, 4*). The curve numbers and film numbers shown in Table 1 are the same.

structure and the metal regions of metal-dielectric composite films, determined by the concentration of metals x.

The results of the conducted studies have shown that in order to obtain maximum values of negative and positive magnetoconductivity at low and high temperatures, it is necessary to use $CoFeB/SiO_2$ composite films with a granular structure and with a small volume of percolation regions, that is, for our studied films with a minimum concentration of metals of 41 at.%. Magnetoconductivity, which is close to zero, is typical for composite films having large volumes with a granular structure and extended metallic regions. Composite films with a metal concentration of $x = 41$ at.%, having low values of static conductivity $0.3 \cdot 10^{-6}\,\Omega^{-1}$ and high values of relative positive magnetoconductivity $+0.2$ at magnetic fields up to 1 T can be used in switching devices. For the use of composite films with high concentrations of metals $x > 64$ at.% in radio and electronic devices, it is necessary to conduct additional studies of the effect of radio pulse and current pulse parameters on changes in magnetoconductivity in various magnetic fields at room temperatures.

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The authors declare that they have no conflict of interest.

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