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Magnetic and Magnetoresistive Properties of Thin Film Alloys Based on Cobalt and Copper

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This work presents the results of a study of the influence of dimensional and concentration effects on magnetoresistive and magnetic properties and structural-phase state of annealed at $T_a = 700$ K samples of thin film alloys based on Co and Cu with thickness $d = 40$ nm in a wide range of total concentrations of Co ($18 \leq c_{Co} \leq 69$ at. %). The samples were obtained by the method of co-evaporation in vacuum using two independent electron guns. It was found that samples of thin film alloys with a total concentration of cobalt $c_{Co} \leq 22$ at. % are characterized mainly by a superparamagnetic state in which hcp-Co granules with size $L = 5 \div 12$ nm were placed in a matrix of Cu(Co) solid solution (s.s.) based on fcc-Cu. The increase in the total concentration of Co in thin film alloys to $c_{Co} = 37$ at. % led to an increase in the size of the magnetic granules to $L = 7 \div 20$ nm and a decrease in the distance between them. In samples of thin film alloys with a cobalt concentration of $56 \leq c_{Co} \leq 69$ at. %, hcp-Co granules with size $L = 8 \div 25$ nm were combined into ferromagnetic clusters, and the volume between the clusters was filled with fcc-Cu(Co) s.s.. The maximum giant magnetoresistance (GMR) values, measured in an external magnetic field $H_{max} = 15$ kOe at room temperature, were obtained for a sample with a total concentration of cobalt $c_{Co} = 22$ at. %. The amplitude of GMR values was 3.8 % and 4.1 % in the perpendicular and longitudinal measurement geometries, respectively. Thin film alloys with a total cobalt concentration of $18 \leq c_{Co} \leq 22$ at. % were characterized by isotropic magnetoresistance. The increase in the total concentration of cobalt in thin film alloys to $c_{Co} \geq 28$ at. % led to the emergence of magnetic anisotropy and anisotropic magnetoresistance. This may be due to the appearance of ferromagnetic interactions between cobalt granules. Low-temperature measurements of magnetoresistance at $T_{meas} = 5$ K of the sample with a total cobalt concentration $c_{Co} = 18$ at. % showed an increase in the amplitude of GMR by 2.1 times (GMR = 11.1 %) compared with the amplitude of GMR = 5.18 % measured at room temperature in an external magnetic field $H_{max} = 90$ kOe. With increasing measurement temperature to $T = 350$ K the amplitude of GMR decreased and amounted to 4.5 %. This is due to the increase in electron scattering by phonons with increasing temperature.

Keywords: Giant magnetoresistance, Solid solution, Spin-dependent scattering, Anisotropy, Coercivity, Superparamagnetism, Granular state.

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1. INTRODUCTION

A lot of attention is paid to the study of magnetic and magnetoresistive properties of thin-film materials and their relationship with the structural-phase state after the discovery of the effect of giant magnetoresistance (GMR) in multilayer films [1] and granular magnetic film alloys [2]. Magnetic film materials which show the effect of GMR are interesting for fundamental research as well as applications [3-6]. Such nanostructures can be used for application in nanoelectronics and spintronics in the manufacture of elements of nonvolatile magnetic memory [3], sensors of magnitude and direction of a magnetic field [7]. In automotive and consumer electronics, such position sensors are attractive because of the non-contact mode of operation and the absence of mechanical interaction between moving parts [3]. In biotechnology, magnetic nanostructures are used in manufacturing biosensors [3] and production of devices for bimolecular detection of proteins in analyzes using magnetic labels [3, 5].

Examples of granular film systems are alloys based on Co and Ag, Co and Cu, Fe and Ag [8]. In [9], it was shown that the GMR effect refers exclusively to systems based on transition 3d metals. Granules of a magnetic component ranging from one to tens of nanometers are placed in a nonmagnetic matrix [10], in granular magnetic films. GMR occurs when the sample is magnetically inhomogeneous, and the distance between the granules is proportional to the mean free path of electrons [11]. The authors [12, 13] showed that the thickness of granular films affects their structural state. Subsequently, the magnetic and magnetoresistive properties of granular film systems depend on the structural state of the film sample: the size and shape of the granules and the distance between them [8, 14].

In the literature, magnetic and magnetoresistive properties of granular systems based on Co and Cu with a thickness $d \geq 100$ nm have been studied in sufficient detail, see, for example, [15]. However, one of the parameters for miniaturizing films as sensitive elements of devices is their thickness. The decrease in the thickness of granular films causes dimensional effects that affect the structure of the samples and, accordingly, the magnetic and magnetoresistive properties. In [4, 13], it was shown that in the case of relatively small ($d \leq 40$ nm) total film thickness, the granular system has some peculiar properties. In a film alloy based on Co and Cu at small thicknesses of the film system, the islands of the magnetic component will act as granules, which will be localized between the crystallites of the fcc-Cu(Co) solid solution (s.s.). In [11], it was shown that when the thickness of the granular film decreases, the threshold of structural percolation and maximum coercivity shifts towards higher concentrations of the magnetic component. There are almost no published results of complex studies of the structural-phase state, magnetic and magnetoresistive properties of granular films based on

Co and Cu with a thickness $d \le 50$ nm in a wide range of concentrations. In this context, the aim of our work is to perform comprehensive experimental research of the influence of dimensional and concentration effects on magnetoresistive and magnetic properties of heat-treated at $T_a = 700$ K thin-film granular alloys based on cobalt and copper with a thickness of $d = 40$ nm in combination with structural and phase studies.

2. EXPERIMENTAL DETAILS

The samples of film alloys based on Cu and Co with a thickness of 40 nm with a total concentration of Co $18 \le c_{Co} \le 69$ at. % were obtained by electron beam evaporation in vacuum at a pressure of 10^{-4} Pa. The films were applied to polished sitall substrates at room temperature by simultaneous evaporation of Cu and Co from two independent evaporators. The thickness of the film samples during the deposition was monitored by the quartz resonator method [16]. The composition of the films was calculated by the method described in [17]. To control the composition of the films, the SEM method (Tescan VEGA) with an EDS analyzer was additionally used. The deviation of the obtained experimental values of the concentration of the components from the calculated values did not exceed 2 at. %, which is within the measurement uncertainty. The annealing of the samples was carried out in vacuum at 10^{-4} Pa in the horizontal tube furnace (Carbolite STF 16/180) at the temperature $T_a = 700$ K. The annealing time was $t = 60$ min.

The crystal structure and phase state were examined by TEM-125K transmission electron microscope. Samples for TEM were obtained by deposition of thin films on TEM grids coated with carbon film. Measurements of the magnetization of film alloys were performed on a vibrating magnetometer at room temperature in the field range $H_{max} = \pm 16.5$ kOe in two measurement setups: "out of plane" (the magnetic field is applied perpendicular to the plane of the sample) and "in-plane" (the magnetic field is applied parallel to the plane of the sample). The measurement of the dependence of the magnetic moment on the measurement temperature in the ZFC-FC modes was performed on a magnetometer MPMS XL SQUID ($T_{meas} = 2 \div 350$ K, $H = 100$ Oe).

The magnetoresistance was measured on an automatic measuring system in a magnetic field up to $H_{max} = \pm 15$ kOe at room temperature using a 4-probe measurement method. Resistance measurements were performed in two different geometries. In the first case, the applied field H was in the plane of the film and parallel to the direction of the current (longitudinal measurement geometry) [15]. In the second case, the applied magnetic field was perpendicular to the plane of the film and to the direction of the current (perpendicular geometry). The current in the plane of the film was 1 mA. Low-temperature measurements of the magnetoresistance were performed on a PPMS Model 6000 from Quantum Design ($T_{meas} = 2 \div 400$ K, $H_{max} = 90$ kOe).

The magnitude of the magnetoresistance was calculated by the expression [18]:

$$\mathrm{GMR} = \Delta R/R(0) = (R(H) - R(0))/R(0), \qquad (1)$$

where $R(H)$ and $R(0)$ are respectively the resistances in an arbitrary magnetic field H and without a magnetic field ($H = 0$).

The coefficient of anisotropic magnetoresistance was calculated by the expression [19]:

$$\mathrm{AMR}\ (\%) = \Delta\rho/\rho_{av} \times 100 = \frac{\rho_S^{\mathrm{II}} - \rho_S^{\perp}}{\left(1/3\rho_S^{\mathrm{II}} + 2/3\rho_S^{\perp}\right)} \times 100, \qquad (2)$$

where ρ_S^{II} and $\rho_S^{\perp}$ are the resistivities at magnetic saturation (I parallel (II) and perpendicular ($\perp$)).

3. RESULTS AND DISCUSSION

3.1 Magnetoresistive and Magnetic Properties

In [20], it was shown that heat treatment of film samples is necessary for homogenization of granular solid solution. In addition, the optimal size of the granules, at which the granular film will have the maximum value of GMR [20], can be achieved by heat treatment. This can be attained by choosing the temperature and annealing time. A series of samples of film alloys investigated in this work were heat-treated at $T_a = 700$ K for a time $t = 60$ min.

Fig. 1 shows the field dependences of the magnetoresistance of film alloys based on Co and Cu measured in perpendicular and longitudinal geometries at room temperature.

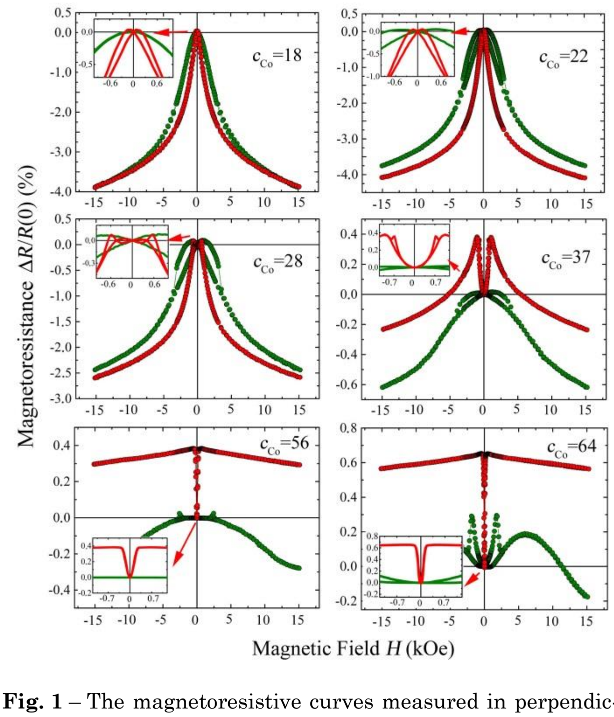

Fig. 1 – The magnetoresistive curves measured in perpendicular (●) and longitudinal (●) geometries at room temperature for the granular thin film alloys based on Co and Cu with a different total concentration of Co c_{Co} (at. %)

As can be seen from Fig. 1, at $c_{Co} \le 22$ at. % the magnetoresistance curves are not saturated in the external magnetic field $H_{max} = \pm 15$ kOe. The absence of saturation on the magnetoresistance curve can be caused by the fact that the structure of the film alloys consists mainly of superparamagnetic (SPM) granules.

A system of non-interacting magnetic granules is characterized by SPM behavior in the case when the internal energy of the ferromagnetic interaction is much less than the thermal energy $kV << k_BT$ (where k is the anisotropy constant, V is the volume of the granule, k_B is the Boltzmann constant). It should be noted that film alloys of this composition are magnetically isotropic. Therefore, the behavior of the magnetoresistance curves obtained in the longitudinal and perpendicular measurement geometries has a similar character. The value of GMR decreases and anisotropic magnetoresistance (AMR) appears at a total concentration of Co $c_{Co} = 28$ at. %. This can be explained by the fact that an increase in cobalt concentration entails an increase in the size of the magnetic granules and a decrease in the distance between them. At the same time, the dipole interaction between the neighboring granules becomes stronger. Fig. 2 shows the dependence of the AMR coefficient of a film alloy based on Co and Cu on the total concentration of Co. The AMR ratio was calculated by expression (2). As can be seen from Fig. 2, an increase in the total concentration of Co in the film alloy leads to an increase in the AMR coefficient.

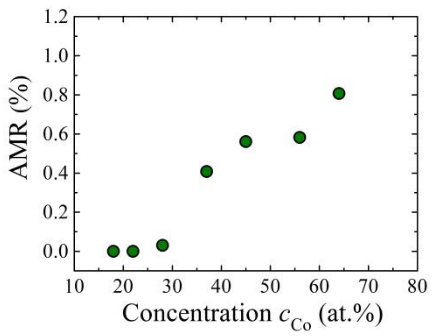

Fig. 2 – The dependence of the coefficient of anisotropic magnetoresistance in thin film alloys based on Co and Cu on the total concentration of Co c_{Co} (at. %). The measurements were carried out at room temperature

Fig. 3 shows the dependence of the GMR value of film alloys in a wide range of composition ($18 \leq c_{Co} \leq 69$).

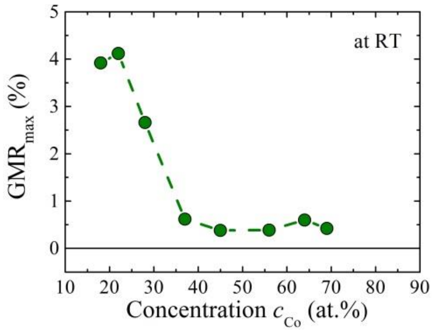

Fig. 3 – Magnetoresistance in the longitudinal geometry of thin film alloys based on Co and Cu as a function of the total concentration of Co c_{Co}. The measurements were carried out at room temperature in magnetic field $H_{max} = 15$ kOe

The study of magnetoresistance was performed in the longitudinal geometry at room temperature in an external magnetic field $H_{max} = 15$ kOe. The value of GMR was determined by the expression (1). The maximum amplitude of GMR = 4.1 % was measured in the sample of a film alloy with cobalt concentration $c_{Co} = 22$ at. %. Samples of film alloys with $c_{Co} \geq 37$ at. % had low values of magnetoresistance. This result is in good agreement with the studies of magnetoresistive properties of film alloys based on Co and Cu given in [22].

It should be noted that the largest number of works [12, 21, 23] on studies of the effect of GMR is focused on film alloys with a low concentration of the magnetic component. In such films, there is a great variety of magnetic and transport phenomena. Fig. 4 shows the field dependences of the magnetoresistance for a film alloy with $c_{Co} = 18$ at. % measured in the longitudinal geometry at temperatures of 5, 300 and 350 K in an external magnetic field $H_{max} = 90$ kOe. At the measurement temperature $T = 5$ K, the amplitude of GMR was 11.1 %. With increasing measurement temperature, the value of GMR decreased to 5.18 % and 4.5 % at $T = 300$ K and 350 K, respectively. This can be explained by the increase in electron scattering by phonons with increasing temperature. As it can be seen from Fig. 4, the magnetoresistance curve obtained at 300 and 350 K is not saturated in the external magnetic field $H = 90$ kOe which indicates the SPM state of this sample.

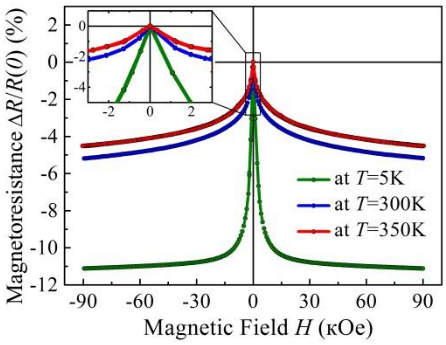

Fig. 4 – Magnetoresistance curves measured in the longitudinal geometry at 5, 300 and 350 K for a granular thin film alloy based on Co and Cu with $c_{Co} = 18$ at. %

Fig. 5 shows the temperature dependence of the magnetic moment M obtained during cooling the sample of a film alloy with $c_{Co} = 22$ at. % in a magnetic field $H = 100$ Oe (FC curve) and following heating without a magnetic field (ZFC curve). This dependence was obtained by the SQUID method. The point of divergence of the ZFC and FC curves determines the maximum blocking temperature [2] ($T_B \approx 225$ K) for a measured film sample. This temperature defines the energy of magnetic anisotropy. As it can be seen from Fig. 5, a granular film alloy based on Co and Cu with a concentration of Co $c_{Co} = 22$ at. % at measurement temperatures $T > T_B$ demonstrates SPM behavior. On the contrary, at $T < T_B$, the granular film alloy is characterized by a ferromagnetic state. The presence of a wide transition on the ZFC-FC curves near the blocking temperature T_B indicates that the size of Co granules in this sample varies in a wide range. Therefore, the granules are in both states: single-domain SPM and single-domain ferromagnetic (SD-FM).

Fig. 6 shows the results of studies of the magnetic properties of the samples of film alloys based on Co and Cu.

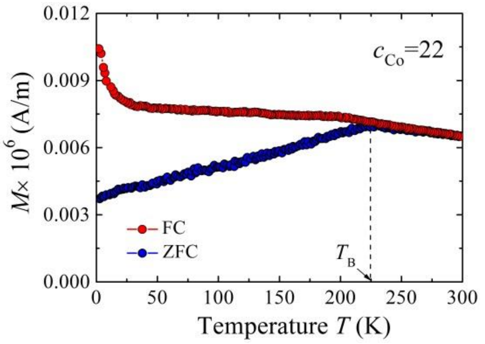

Fig. 5 – ZFC/FC curves for a granular thin film alloy based on Co and Cu with c_{Co} = 22 at. %. The measurements were carried out for geometries of the magnetic field applied "in plane" at H = 100 Oe

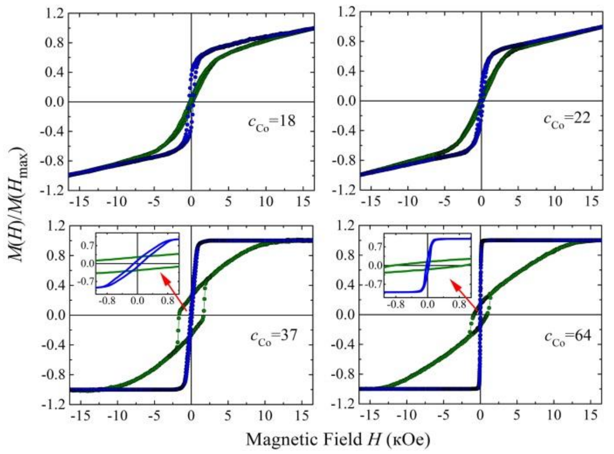

Fig. 6 – Hysteresis loops for granular thin film alloys based on Co and Cu with a different total concentration of Co for geometries of the magnetic field applied "in-plane" (●) and "out of plane" (●). The measurements were carried out at room temperature

The field dependences of the magnetization of Co- and Cu-based film alloys with different total Co concentrations were measured in "in-plane" and "out-of-plane" geometries at room temperature. As it can be seen from Fig. 6, the behavior of the magnetization curves depends on the composition of the film sample. At small values of the total concentration of Co in the film alloy (at $c_{Co} \leq 22$ at. %), the curves are not saturated in the external magnetic field $H_{max} = \pm 16.5$ kOe in both geometries. Such dependences are characteristic of samples, the structure of which consists mainly of SPM granules. As the concentration of cobalt in the alloy increases to c_{Co} = 37 at. %, the samples become magnetically anisotropic. This is confirmed by the different behavior of hysteresis loops for "in-plane" and "out of plane" geometries. The main reason for the appearance of anisotropy may be the magnetic interaction between the magnetic granules of cobalt. Granular film systems, characterized by the AMR effect, are in the focus of interest from a practical point of view in the manufacture of sensor elements for measuring the angle of rotation of the object [3, 7]. These sensors are widely used in the automotive industry [3]. For this purpose, we made a comprehensive study of the magnetic and magnetoresistive properties of a sample of a film alloy with a total concentration of Co c_{Co} = 37 at. % when rotating it in an external magnetic field from parallel to perpendicular geometry. Position 0° corresponded to the longitudinal (magnetoresistance measurement) and "in-plane" geometries (magnetization measurement), and position 90° – to the perpendicular and "out of plane" geometries, respectively. As it can be seen from Fig. 7, the sample rotation in the external magnetic field changes the shape of the magnetoresistance curve and the hysteresis loop.

Magnetoresistance curves (Fig. 7a) show that switching the sample from the longitudinal geometry position to the perpendicular geometry position resulted in a transition from anisotropic to isotropic magnetoresistance. When the sample was rotated from "in-plane" to "out of plane" geometry (Fig. 7b), the coercivity of the sample increased from H_c = 0.27 kOe to H_c = 1.65 kOe (see the insert in Fig. 7b). The saturation magnetization also increased from H = 0.5 kOe to H = 12.4 kOe.

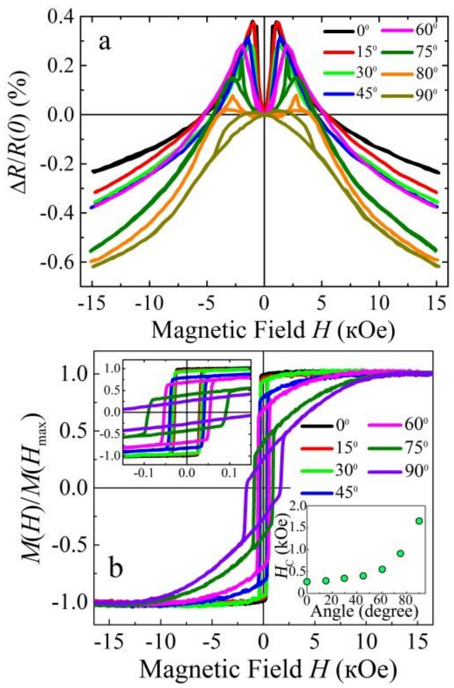

Fig. 7 – The magnetoresistance curves (a) and hysteresis loops (b) for a granular thin film alloy based on Co and Cu with c_{Co} = 37 at. %. The measurements were performed at room temperature. 0^0 corresponds to the longitudinal (a) and "in-plane" (b) geometries, 90^0 corresponds to the perpendicular (a) and "out of plane" (b) geometries

3.2 Structural-phase State

The study of the structural-phase state of annealed at T_a = 700 K film alloys based on Co and Cu in the range of $18 \leq c_{Co} \leq 69$ at. % was carried out by the TEM method. Fig. 8 and Fig. 9 show, respectively, the diffraction patterns and the diffraction spectrum of samples of thin film alloys based on Cu and Co with different total concentrations of Co. The vertical lines in Fig. 9 indicate the tabular values of the diffraction lines for massive samples of fcc-Cu and hcp-Co.

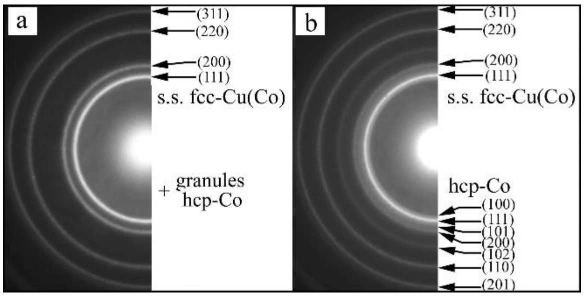

Fig. 8 – The diffraction patterns of alloy thin films based on Co and Cu with a total concentration of c_{Co} = 22 at. % (a) and c_{Co} = 69 at. % (b)

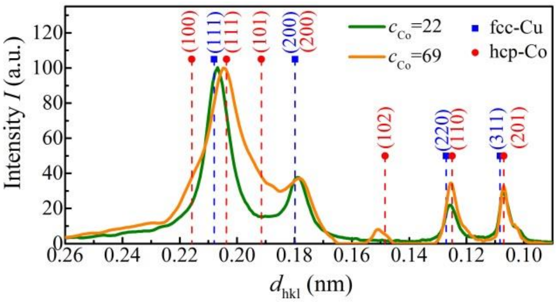

Fig. 9 – Diffraction spectra from samples of thin film alloys based on Co and Cu with a total concentration of c_{Co} = 22 at. % and c_{Co} = 69 at. %

Fig. 10 shows TEM images of the structural state of alloy thin films based on Co and Cu with a different total concentration of Co.

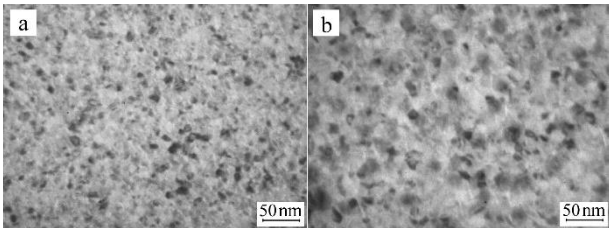

Fig. 10 – The TEM micrographs of thin film alloys based on Co and Cu with a total concentration of Co c_{Co} = 22 at. % (a) and c_{Co} = 69 at. % (b)

Samples of the film alloy with a low total concentration of cobalt (c_{Co} = 22 at. %) were characterized by a state in which hcp-Co granules of size L = 5÷12 nm (Fig. 10a) are included in a matrix of dilute fcc-Cu(Co) s.s. At mentioned sizes, cobalt granules are mainly in the SPM state. An increase in the total concentration of Co to c_{Co} = 37 at. % caused an increase in the size of Co granules to L = 7÷20 nm. Obviously, the distance between the granules became shorter. However, the phase composition of the samples of film alloys did not change. At the total concentration of cobalt c_{Co} = 69 at. % there was a further increase in the size of hcp-Co granules to L = 8÷25 nm (Fig. 10b) and their combination into ferromagnetic clusters. The gaps between the clusters were filled with fcc-Cu(Co) s.s.

The results of structural-phase studies presented in this paper are consistent with the results of studies performed by the authors [16] for samples of film alloys based on Co and Cu with a thickness d = 20 nm in the range of total concentrations of Co 15 ≤ c_{Co} ≤ 71 at. %.

4. CONCLUSIONS

1. The influence of the composition on magnetoresistive and magnetic properties, as well as the structural-phase state of samples of thin film alloys based on Co and Cu with a thickness d = 40 nm, heat-treated at T_a = 700 K, was experimentally established.

2. It was found out that samples of thin film alloys with a total concentration of cobalt c_{Co} ≤ 22 at. % are characterized by a state in which SPM and SD-FM hcp-Co granules with a size L = 5÷12 nm are placed in the matrix of fcc-Cu(Co) s.s. An increase in the total concentration of Co in the film alloy to c_{Co} = 37 % led to an increase in the size of the magnetic granules to L = 7÷20 nm and a decrease in the distance between them. As a result, the dipole interactions between adjacent Co granules became more significant. In the samples of film alloys with a cobalt concentration of 56 ≤ c_{Co} ≤ 69 at. %, the hcp-Co granules with size L = 8÷25 nm were combined into ferromagnetic clusters, and the volume between the clusters was filled with fcc-Cu(Co) s.s.

3. The maximum values of GMR at room temperature in an external magnetic field H_{max} = 15 kOe were obtained for a sample with a total concentration of cobalt c_{Co} = 22 at. %. The values of GMR were 3.8 % and 4.1 % in the perpendicular and longitudinal measurement geometries, respectively. At total concentrations of cobalt c_{Co} ≥ 28 at. % the samples were characterized by AMR.

4. Low-temperature measurements of magnetoresistance at T_{meas} = 5 K of a sample of a film alloy with a total concentration of cobalt c_{Co} = 18 at. % showed an increase in the amplitude of GMR by 2.1 times up to 11.1 % in the field H_{max} = 90 kOe compared with the amplitude of GMR = 5.18 % measured at room temperature. An increase in the measurement temperature to T = 350 K decreased the amplitude of GMR down to 4.5 %, that can be caused by an increase in electron scattering by phonons with increasing temperature.

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Магнітні та магніторезистивні властивості тонкоплівкових сплавів на основі кобальту і міді

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У даній роботі представлені результати дослідження впливу розмірного та концентраційного ефектів на структурно-фазовий стан, магніторезистивні та магнітні властивості відпалених при $T_B = 700$ К зразків тонкоплівкових сплавів на основі Co і Cu товщиною $d = 40$ нм у широкому діапазоні загальних концентрацій Co ($18 \leq c_{Co} \leq 69$ ат. %). Зразки отримували методом одночасного випаровування у вакуумі з використанням двох незалежних електронних гармат. Встановлено, що зразки тонкоплівкових сплавів із загальною концентрацією кобальту $c_{Co} \leq 22$ ат. % характеризуються переважно суперпарамагнітним станом, при якому гранули ГЩП-Co розміром $L = 5\div12$ нм були розміщені в матриці твердого розчину (т.р.) Cu(Co) на основі ГЦК-Cu. Збільшення загальної концентрації Co у плівковому сплаві до $c_{Co} = 37$ ат. % призвело до збільшення розмірів магнітних гранул до $L = 7\div20$ нм та зменшення відстані між ними. У зразках плівкових сплавів з концентрацією кобальту $56 \leq c_{Co} \leq 69$ ат. % гранули ГЩП-Co розміром $L = 8\div25$ нм об'єднувалися у феромагнітні кластери, а об'єм між кластерами був заповнений т.р. ГЦК-Cu(Co). Максимальні значення ГМО, виміряні у зовнішньому магнітному полі $H_{макс} = 15$ кЕ при кімнатній температурі, було отримано для зразка з загальною концентрацією кобальту $c_{Co} = 22$ ат.%. Амплітуда значень ГМО при цьому становила 3.8 % та 4.1 % у перпендикулярній та поздовжній геометріях вимірювання відповідно. Плівкові сплави з загальною концентрацією кобальту $18 \leq c_{Co} \leq 22$ ат. % характеризувалися ізотропним магнітоопором. Зростання загальної концентрації кобальту у плівковому сплаві до $c_{Co} \geq 28$ ат. % призвело до виникнення магнітної анізотропії та анізотропного магнітоопору. Це може бути пов'язано з появою феромагнітних взаємодій між гранулами кобальту. Низькотемпературні вимірювання магнітоопору при $T_{вим} = 5$ К зразка плівкового сплаву з загальною концентрацією кобальту $c_{Co} = 18$ ат. % показали зростання амплітуди ГМО в 2,1 рази (ГМО = 11.1 %) порівняно з амплітудою ГМО = 5.18 %, виміряною при кімнатній температурі у зовнішньому магнітному полі $H_{макс} = 90$ кЕ. При збільшенні температури вимірювання до $T_{вим} = 350$ К амплітуда ГМО зменшилася і становила 4,5 %. Це пов'язано із збільшенням розсіювання електронів на фононах з ростом температури.

Ключові слова: Гігантський магнітоопір, Твердий розчин, Спін-залежне розсіювання, Анізотропія, Коерцитивність, Суперпарамагнетизм, Гранульований стан.