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Study of the Effect of the Chemical Composition of Thin Nanostructured Films of Cu-Ni Alloys on the Structure, Electrical and Magnetic Properties

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***Abstract* — The results of a study of the effect of the chemical composition of thin nanostructured films of Cu-Ni alloys on their crystalline structure, electrical resistance, thermal coefficient of resistance and galvanomagnetic properties are presented.**

***Keywords* — nanostructured films, alloy films, chemical composition, crystal structure, electrical conductivity, magnetoresistance**

I. INTRODUCTION

Large-scale study of the physical properties of thin metal films has led to the emergence in recent decades of a new branch of solid-state physics — thin film physics. Advances in the thin film physics have found wide use in nanoelectronics, high-frequency technology, optoelectronics, optics and many other areas of modern science and technology.

Films of magnetically ordered metals Ni, Fe, Co and alloys based on them occupy a special place in the physics of thin films: solving the fundamental problems of “two-dimensional magnetism”, giant magnetoresistance, spin-polarized tunneling, colossal magnetoresistance, etc. These discoveries are widely used in nanoelectronics and spintronics for the development of miniature magnetoelectronic devices, new methods for recording and storing information, and new types of highly sensitive sensors and transducers [1-8].

Unlike well-developed technologies for producing massive alloys, technologies for producing film alloys still remain not fully studied. A feature of technologies for producing film alloys in vacuum is the appearance of some uncertainty in the formation of a sample with a predetermined chemical composition. Studies of the chemical composition of film alloys using methods that are effective for massive alloys cause certain difficulties due to the extremely small amount of the analyzed substance. The modern most widely used methods for studying the chemical composition of film alloys are X-ray spectral microanalysis and secondary ion mass spectrometric microanalysis. Both methods have their advantages and disadvantages.

This paper presents the results of a study of the effect of the chemical composition of thin nanostructured films of Cu-Ni alloys on their crystalline structure, electrical resistance, thermal coefficient of resistance and galvanomagnetic properties.

II. EXSPERIMENTAL DETAILS

Films of Cu-Ni alloys with thicknesses of 1-200 nm were obtained by condensation of the starting materials (Cu and Ni) with their simultaneous evaporation from two independent evaporators according to the Vekshinsky method. This made it possible to obtain films of an alloy of arbitrary composition on the substrate. For copper, the method of thermal evaporation of Cu from a tungsten foil strip 0.05 mm thick was used. For more refractory nickel, the method of electron beam evaporation of Ni using an electron diode gun was used. The purity of the evaporated metals was no less than 99.98%. The condensation rate was determined from the condensation time and the final thickness of the sample. During the condensation process, it remained constant and amounted to 0.5-1.5 nm/s.

The design of the substrate holder we manufactured made it possible to obtain four film samples of different concentrations of components with almost the same thickness in one sputtering technological cycle.

Polished optical glasses with pre-applied contact pads (study of electrical and magnetoresistive properties), NaCl single crystals and carbon films (structural studies) were used as substrates [9].

The film thickness was measured using a MII-4 microinterferometer we modernized with a laser light source and digital processing of the interference pattern. As a result, it is possible to reduce the error in measuring film thickness, especially in the thickness range $d < 50$ nm. The thickness measurement error was 5-10% for thicknesses 50-200 nm and 10-15% for thicknesses $d < 50$ nm.

To determine the concentration of alloy film components, the calculation method and the X-ray microanalysis method were used.

The essence of the calculation method is illustrated by the diagram presented in Fig. 1.

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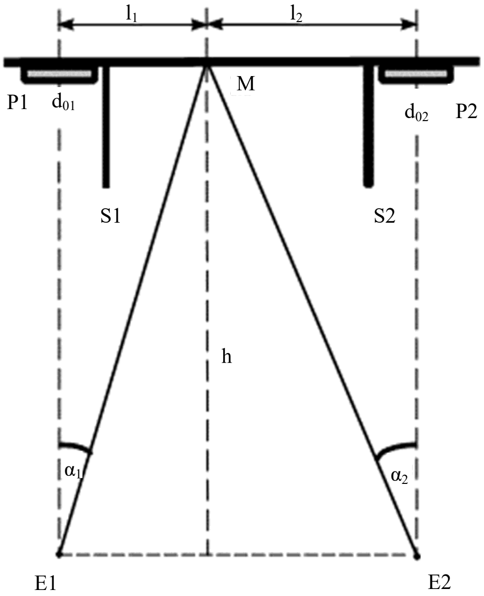

Fig. 1. Geometry of the «substrate-evaporators» system for calculating the concentration of the alloy components: S1, S2 – screens; E1, E2 – evaporators; P1, P2 – glass plates “witnesses” of thickness.

Based on the geometry of the “substrate-evaporators” system, the thickness of individual components d_1 and d_2 at the point M of the substrate can be calculated from the relations [10]:

$$d_1 = d_{01}\left[1+\left(\frac{l_1}{h}\right)^2\right]^{-\frac{3}{2}} \text{ and } d_2 = d_{02}\left[1+\left(\frac{l_2}{h}\right)^2\right]^{-\frac{3}{2}}, \quad (1)$$

where d_0 – the film thickness at the point above the evaporator; d_{01}, d_{02} – the thicknesses of the “witness” films; l_1, l_2 – the distance from the middle of the “witness” to the point M; h – the distance from the plane of the evaporators to the plane of the substrate.

To find the concentration of the C_1 and C_2 components of the alloy at the M point of the substrate, one can use the obvious relations:

$$C_1 = \frac{D_1 d_1 \mu_1^{-1}}{D_1 d_1 \mu_1^{-1} + D_2 d_2 \mu_2^{-1}}, \; C_2 = \frac{D_2 d_2 \mu_2^{-1}}{D_1 d_1 \mu_1^{-1} + D_2 d_2 \mu_2^{-1}}, \quad (2)$$

where D_1 and D_2 are the film densities of each of the materials; μ_1 and μ_2 are the molar masses of evaporated metals.

Taking into account that in bulk samples $D_{Ni} \approx D_{Cu}$, ($D_{Ni} = 8.897$ g/cm^3, $D_{Cu} = 8.932$ g/cm^3 [11]) and assuming that the same is true for films, relations (2) are trans-formed to the form:

$$C_1 = \frac{d_1 \mu_2}{d_1 \mu_2 + d_2 \mu_1} \quad \text{and} \quad C_2 = \frac{d_2 \mu_1}{d_1 \mu_2 + d_2 \mu_1}. \quad (2')$$

Since the concentration of the alloy components was determined by the thicknesses d_1 and d_2, the error in determining the concentration of the components is the same as for the thickness.

The study of the elemental composition of thin films of the Cu-Ni alloy was carried out using an X-ray microanalyzer based on an energy dispersive spectrometer (EDS) included in the REM-103-01 scanning electron microscope. The characteristic X-ray radiation of the atoms of the elements of the sample was measured using the EDS with subsequent processing of the information using the software of the X-ray microanalyzer. The EDS scheme is shown in Fig. 2.

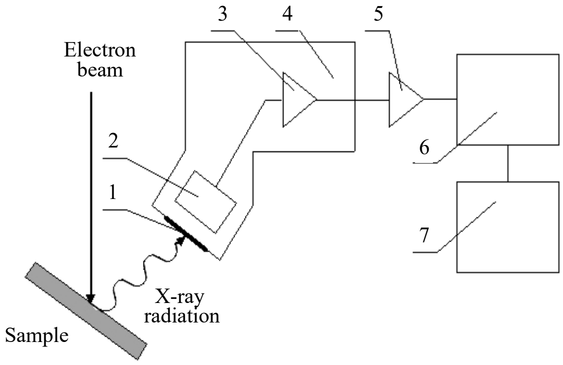

Fig. 2. Scheme of the spectrometer with EDS.

The characteristic X-ray radiation, which is generated in the sample when its surface is probed with a high-energy electron beam, passes through a thin beryllium window (1) into the cryostat (4), where a cooled (to reduce the noise level) detector (2) is located in vacuum. Further, the signal is amplified in amplifiers (3) and (5) and enters the multichannel pulse analyzer (6). Information is extracted from it using special computer software (7) and presented in the form of a spectrum of dependence of the number of pulses (X-ray intensity) on the energy of X-ray photons. Methods of X-ray spectral analysis using EDS can determine the concentration of almost any element from beryllium to californium in the concentration range up to tenths of an atomic percent.

Since the amount of substance in the film samples studied by us is extremely small, especially for films with effective thicknesses d ~ 10 nm, the characteristic X-ray spectrum of the film substance was excited by scanning a film section with dimensions of 300×300 μm with an electron beam. This made it possible to obtain integrated data on the elemental composition of the film without overheating it with an electron beam. For thicker films, it was possible to reduce the size of the film section to 1×1 μm.

Although the theory of quantitative elemental X-ray spectral analysis of thin films is based on the same prerequisites as for bulk samples (model ideas about the physical processes of the interaction of electrons with matter), the X-ray spectral microanalysis of thin films has its own characteristics due to differences in the absorption and transmission of X-rays in bulk samples and samples in the form of thin films.

In quantitative measurements of the elemental composition of thin films of binary Cu-Ni alloys, we used a thin Ni film as a standard in accordance with the recommendations set forth in [12]. Nickel is included in the alloy films under study, and the densities of copper and nickel are close.

The study of the galvanomagnetic properties of film alloy samples was carried out in accordance with the methodology described in detail in [13].

The calculation method and X-ray microanalysis were used to determine the concentration of the alloy film components. A comparison of the results shows that in the range of sample thicknesses $d < 100$ nm, the discrepancy between the calculated and measured concentration values is about 10%. For sample thicknesses $d > 100$ nm, the discrepancy decreases to 1-3%.

The decrease in the discrepancy between the calculated and experimentally measured results of determining the concentration of the components is fully explained by the fact that with an increase in the film thickness d, measured with the MII-4 microinterferometer and included in relation (2), the relative error of its measurement decreases.

Unfortunately, X-ray microanalysis does not allow obtaining information about the presence of chemical compounds (hydrides, oxides, nitrides, etc.) in the samples, the formation of which is possible during the condensation of samples during the interaction of the film material with gases of the residual atmosphere (H_2, O_2, N_2, etc.) as a result of their sorption on the film surface. The presence of such compounds can significantly affect the physical properties of film samples (electrical conductivity, phase composition, etc.). The detailed results of measurements of the chemical composition of films of Cu-Ni alloys are presented in [14, 15].

III. RESULTS AND DISCUSSION

A. Dependence of fcc lattice parameter of alloy films on component concentration

Cu-Ni film alloys in all thickness ranges d = 1-200 nm and concentrations $0 < C_{Cu} < 100$ at.% studied by us have an fcc lattice with a parameter that depends on the concentration of the components and varies from $a = 0.352$ nm up to $a = 0.362$ nm. These results are in good agreement with literature data for bulk alloys ($0.3524 < a_0 < 0.3615$ nm) [16].

It should be noted that the formation of the fcc alloy occurs already at the condensation stage, which is confirmed by electron diffraction (Fig. 3).

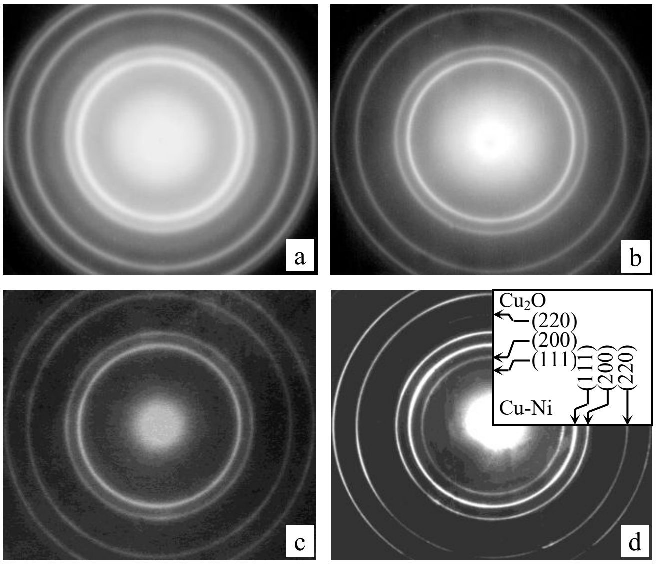

Fig. 3. Electron diffraction images of unannealed (a, c) and annealed to 700 K (b, d) Cu-Ni alloy films: $d \approx 1$ nm, $C_{Cu} = 50.3$ at.% (a, b); d = 40 nm, $C_{Cu} = 38$ at.% (c, d).

As can be seen from Fig. 3, for unannealed films in the entire range of thicknesses and concentrations, the first line (111) is very wide. In this case, one gets the impression that it is double (this would take place in the case of the eutectic structure of the fcc Cu + fcc Ni alloy), but this is not so. The difference $\Delta d_{111} = d_{111}(Cu) - d_{111}(Ni) = 0.009$ nm is quite sufficient to observe the (111)Cu and (111)Ni lines separately by electron diffraction.

Electron diffraction patterns from thermally stabilized structurally layered samples (Fig. 3 d) sometimes show additional extremely thin lines (from 2 to 5 lines) of Cu_2O oxide, which has an fcc lattice with a parameter a = 0.424-0.450 nm. It should be noted that the two most intense lines $(111)Cu_2O$ and $(220)Cu_2O$ are observed in electron diffraction patterns from freshly condensed films at a condensation rate of $\omega < 0.5$ nm/s. At the same time, it should be noted that under our experimental conditions no lines of unstable oxide NiO or stable nitride Ni_3N were observed, although a number of authors noted this phase formation in nickel films.

The results of studying the dependence of the lattice parameter of the Cu-Ni film alloy on the concentration of the components are shown in Fig. 4. We note the fact that the lattice parameter in alloy films is somewhat larger compared to bulk samples. This increase can be partly explained by the penetration of atoms from the residual atmosphere into the crystal lattice of the alloy, and partly by the fact that the atoms of one of the alloy components during condensation can occupy positions that do not correspond to an ordered alloy.

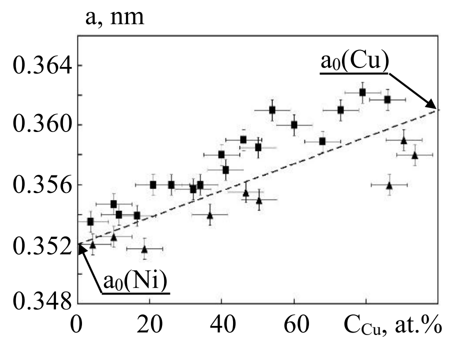

Fig. 4. Concentration dependence of the fcc lattice parameter of Cu-Ni alloys (measurement temperature T = 300 K): ■ – continuous films; ▲ – island films; --- – Vegard's rule.

As for bulk samples (dashed line in Fig. 4), for Cu-Ni alloy films, an increase in the values of the lattice parameter from $a = 0.353$ nm to $a = 0.362$ nm is observed. For ultrathin Cu-Ni alloy samples with effective thicknesses d = 1-7 nm, the crystal lattice parameter is 0.002-0.003 nm less compared to bulk samples. There are a large number of experimental works in the literature (see, for example, [17, 18]) in which it was shown that in island films of pure fcc metals (including Ni and Cu, which make up the alloys under study), depending on the production conditions, the parameter lattice parameter can be either smaller or larger than the bulk metal lattice parameter, increasing or decreasing with increasing island size. However, under conditions where the influence of gas impurities is minimized [17], the lattice parameter always decreases with decreasing particle size.

B. Concentration effects in electrical conductivity

When studying the electrical conductivity of the films, we measured the electrical resistivity ρ and the thermal coefficient of resistance (TCR) β. For ultrathin samples of Cu-Ni alloy films of various concentrations of components, having an island structure with effective thicknesses d = 1-

2 nm, we observed anomalous ($\beta < 0$) TCR values. For structurally continuous Cu-Ni films that underwent thermal stabilizing annealing up to 700 K, in all studied ranges of thicknesses and component concentrations, only positive values of the temperature coefficient of resistance ($\beta > 0$) were observed, while the TCR value was the same as for bulk Cu-Ni alloys, depended on the concentration of the components (the tendency of the concentration dependence of β to pass through a minimum remained). It should be noted that our experimental TCR values for Cu-Ni film alloys generally slightly exceed the β_0 values for bulk samples (Fig. 5 a), although $\beta < \beta_0$ is always observed in single-component films. One explanation for this fact may be that the TCR for massive samples, as a rule, is presented for structurally equilibrium alloys.

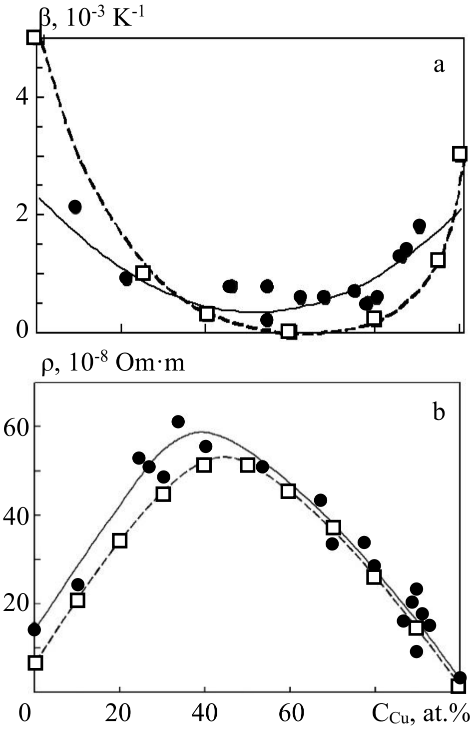

Fig. 5. Concentration dependence of TCR (a) and ρ (b) of Cu-Ni film alloys (●): □ – data for bulk alloys [11].

In Fig. 5 b shows the dependence of the resistivity of the studied film alloys with a thickness d = 80-125 nm on the concentration of the components. As can be seen from the figure, the tendency of the concentration dependence of resistivity to pass through the maximum remains, although the maximum is shifted by 10 at.% towards a decrease in copper concentration. It should be noted that our results are in good agreement with literature data [19] for electrodeposited Ni-Cu alloys and micron thicknesses.

The experimental resistivity values of Cu-Ni film alloys generally exceed the resistivity values in bulk samples, which can be explained by the manifestation of size effects.

C. Concentration effects in galvanomagnetic properties

For all samples with concentrations $C_{Cu} < 35$ at.%, magnetoresistance (MR) hysteresis was observed. When the direction of the magnetic field was reversed, corresponding hysteresis loops in the $\Delta R(H)/R_0$ dependence were observed, which suggests the presence of a domain structure in such films. However, at $C_{Cu} > 35$ at.% the MR effect is not observed at room temperature. This can be explained by the fact that at such concentrations of Cu in samples of Cu-Ni alloys, the Curie point lies below room temperature [11].

Annealing films without the influence of an external magnetic field leads to a more clear manifestation of hysteresis phenomena of magnetoresistance. In this case, the value of $\Delta R_{max}/R_0$ is somewhat larger, which is obviously due to the improvement of the domain structure during the annealing process due to an increase in the size of crystallites, a decrease in the number of defects in the crystal structure, and stress relief.

Annealing of films in an external magnetic field of strength H = 8 kA/m leads to a more clear manifestation of hysteresis phenomena of magnetoresistance. In this case, the value of $\Delta R_{max}/R_0$ reaches large values (for example, for samples with C_{Cu} = 12-15 at.% at room temperature measurements, the value of $\Delta R_{max}/R_0$ is 0.7% versus 0.2-0.3%), and saturation of the $\Delta R(H)/R_0$ dependence is achieved in lower fields (H = 4-5 kA/m versus H = 30-50 kA/m). The magnetoresistance hysteresis loops of such samples become more "rectangular", which indicates significant differences in the domain structure of films that have undergone different thermomagnetic treatments.

It should be noted that for all samples with a copper concentration greater than 35 at.%, even at a temperature T = 150 K, the MR effect was practically not observed, although, according to literature data [11], alloys with C_{Cu} = 35-45 at.% at low temperature measurements should be ferromagnetic (Θ_{0C} = 270-150 K).

According to [20], in films formed in the absence of an external magnetic field, the domains have the shape of irregular wedges. The process of magnetization reversal of such films occurs through the growth of energetically favorable "old" and the appearance of "new" wedge-like domains, until the new domain phase fills the entire volume of the film (while island-wedges of the previous magnetic phase remain at the edges of the film). The magnetic hysteresis loop of such films is inclined, and the suppression of the edge wedge-shaped domains by an external field leads to an increase in the slope of the loop.

In films that have undergone heat treatment in a magnetic field, the domain structure is much simpler [20]: the domains have the form of stripes oriented along and against the direction of the external field. The processes of magnetization reversal in such films occur by increasing the width of energetically favorable domains (by displacing domain walls) until the new domain phase fills the entire volume of the film. A small number of wedge-shaped domains at the edges of such films leads to the fact that the shape of the magnetic hysteresis loop is close to rectangular, and the magnetization reversal process itself occurs abruptly in a narrow range of external magnetic field strengths and at lower values. The manifestation of these magnetization reversal mechanisms obviously affects the shape of the galvanomagnetic hysteresis loops, but we are not aware of any work with detailed studies of this connection.

The features of magnetoresistance due to the presence of a domain structure make it possible to determine the Curie temperature Θ_C of the alloys based on the dependence of the value $\Delta R_{max}/R_0$ on the copper concentration in Cu-Ni film alloys or, by fixing a certain temperature of the MR measurement, to determine the presence of domains in the film. In Fig. 6 shows the dependence of the value $\Delta R_{max}/R_0$ on the concentration of Cu for film alloys at T = 300 K. At copper concentrations $C_{Cu} > 35$ at.%, the value $\Delta R_{max}/R_0$ takes values close to zero (the domain structure of films does not manifest itself in MR at room temperature).

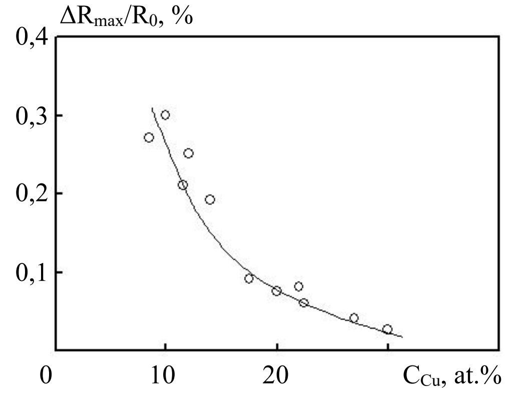

Fig. 6. Concentration dependence of the longitudinal MR value $\Delta R_{max}/R_0$ in films of the Cu-Ni alloy, obtained at a measurement temperature of 300 K.

To determine the Curie temperature Θ_C of Cu-Ni film alloys, we used the method of determining Θ_C from the extremum of the β(T) dependence graph. According to [10], this technique is widely used to determine the Curie point in film samples.

Typical dependences of electrical resistivity ρ and TCR β on temperature for films of Cu-Ni alloys that have undergone stabilizing annealing up to 700 K in vacuum for 3-4 cycles according to the “heating↔cooling” scheme are presented in Fig. 7. For comparison, this figure also shows the dependences ρ(T) and β(T) obtained by us for Ni films.

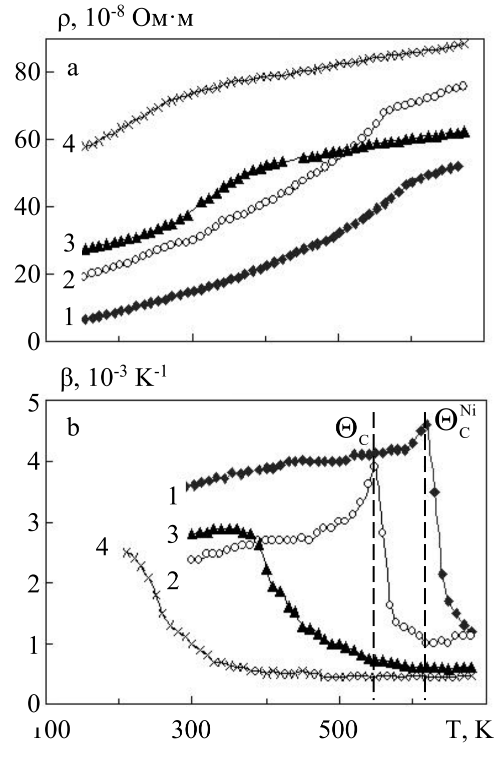

Fig. 7. Dependences of ρ (a) and TCR (b) on temperature for Ni films (1) annealed to 700 K and Cu-Ni alloys (2-4): d = 60 nm (1); d = 65 nm, C_{Cu} = 4 at.% (2); d = 57 nm, C_{Cu} = 14 at.% (3); d = 89 nm, C_{Cu} = 31.6 at.% (4).

The break in the ρ(T) graph observed in Ni films corresponds to the Curie point and is observed at a temperature Θ_C^{Ni} = 620 K (curve 1 in Fig. 7 a), which is in good agreement with the data of other authors. In the β(T) graph, at this point, as for massive samples [21], a sharp maximum is observed (curve 1 in Fig. 7 b). The decrease in value compared to bulk samples (Θ_{0C} = 631 K [11]) is explained by the manifestation of size effects for film samples.

According to literature data [11, 21], for massive samples of Cu-Ni alloys the value of Θ_{0C} decreases linearly with increasing copper concentration up to 90 K at C_{Cu} = 50 at.%. Analysis of experimental data for film alloys with a low copper concentration (C_{Cu} = 4 at.%) shows that in this case the nature of the temperature dependences of resistivity and TCR (curves 2 in Fig. 7 a, b) is similar to the corresponding dependences for Ni films. The decrease in the Curie temperature in alloys of this composition to Θ_C = 550 K is due to concentration (presence of Cu) and size effects (for massive alloy samples with C_{Cu} = 4 at.% the Curie temperature Θ_{0C} = 570 K [11]). A further increase in the copper concentration in film alloys leads to a “blurring” of the maximum in the β(T) dependence graphs (curves 3 and 4 in Fig. 7 b), which in turn does not allow the Curie point to be clearly recorded (for massive samples these values are Θ_{0C} = 398 K and Θ_{0C} = 293 K for C_{Cu} = 14 at.% and C_{Cu} = 31.6 at.%, respectively [11]). Based on the above, we can conclude that in the case of Cu-Ni film alloys, the method of determining the Curie point from the extremum of the graph of the temperature dependence of the TCR can be correctly used only in the case of copper concentrations C_{Cu} < 10-12 at.%.

IV. CONCLUSION

Based on the results of the research, the following conclusions can be drawn:

1. Film alloys of all studied thicknesses and concentrations have an fcc lattice with a parameter that depends on the concentration of the components and varies from a = 0.352 nm to a = 0.362 nm. The obtained dependence of the fcc lattice parameter of alloy films on the concentration of components is generally consistent with Vegard’s rule.
2. Concentration effects in the electrical conductivity and TCR of film alloys have been established. As for massive alloys, the concentration dependence of resistivity is observed to pass through a maximum (for TCR – through a minimum), although both of them are shifted by 10 at.% towards a decrease in copper concentration.
3. It was found that for all samples with a copper content of more than 35 at.%, even at a temperature of T = 150 K, the effect of magnetoresistance is practically not observed, which indicates the absence of a domain structure in them.

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