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LARGE VALUES OF THE TRANSVERSE MAGNETIC RESISTANCE OF SINGLE CRYSTAL NICKEL FILMS

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Abstract:

Background. The anisotropy of the transverse magnetoresistance of single-crystal nickel films was studied in this work. The measurements were carried out on samples whose surface plane coincided with the [001] plane. Studies of the magnetoresistance in a single-crystal nickel film have shown the effect of tensile stresses acting on it from the side of magnesium oxide. The modification

of the anisotropy of magnetoreflexion of a film on a substrate as compared to a free sample is apparently associated with a change in the shape of the Fermi surface of carriers.

In accordance with the technology the films were grown at a substrate temperature of 1000°C. The thermal expansion coefficient of nickel, α_{Ni} , exceeds the corresponding coefficient α_{MgO} for magnesium oxide. For example, at room temperature $\alpha_{Ni} = 12,5 \cdot 10^{-6}$, $\alpha_{MgO} = 11,2 \cdot 10^{-6} \text{ deg}^{-1}$. Consequently, upon cooling, the film, being bonded to the substrate, is subjected to in-plane tensile stress, which leads to tetragonal deformation of the crystal lattice. This conclusion was confirmed by comparing the crystal lattice parameters measured by X-ray diffraction at room temperature T_k and $T = 77 \text{ K}$.

Methods. The magnetoresistance was measured by an unbalanced double Thomson bridge with an accuracy of the order of 10^{-6} Ohm . The magnetoresistance measurements were carried out in the longitudinal and transverse directions of the external magnetic field, as well as depending on the angle φ (between the [100] axis and the direction of the external field). In all measurements the current passed through the samples remained constant $5 \times 10^{-4} \text{ A}$. When measuring the longitudinal and transverse magnetoresistance the external magnetic field varied from 0 to 21000 Oe. The effect measurements in our experiments were carried out with an accuracy of 2-4% on average.

The measurement of the magnetoresistance as a function of the angle φ was carried out at $[100] \parallel i \parallel r \wedge H = \varphi$.

Findings. Oxidation of nickel films occurs through the transfer of electrons, nickel cations and oxygen anions through the film. Annealing controls the oxidation process and film morphology.

According to the results of the analysis of this study we find that Ni films 50 nm thick are almost completely oxidized at an annealing temperature of 700 ° C. It is assumed that tensile density stresses transform the Ni/MgO film into a “strong” ferromagnet, characterized by a high magnitude of magnetoresistance (up to 0.25% at low temperatures). In this work the anisotropy of the transverse magnetoresistance of single-crystal nickel films was studied. The measurements were carried out on samples whose surface plane coincided with the [001] plane. We used samples with a thickness of $d = 5.0 \pm 0.5 \mu\text{m}$ with a ratio $\eta = 10^3$.

Conclusions. The modification of the anisotropy of the magnetoresistance of a film on a substrate as compared to a free sample is apparently associated with a change in the shape of the Fermi surface of carriers. In this case, the volume occupied by carriers in momentum space does not change that is indicated by the existence of a general universal dependence for the magnetoresistance of films on a substrate and films separated from it.

The magnetoresistance in both longitudinal and transverse magnetic fields for all investigated film thicknesses has a different course of curves: in the first case- with a positive value, in the second-with a negative value of the effect magnitude.

The magnitude of the magnetoresistance at a saturation field of 5800 Oe depending on the angle φ between the [100] axis and the direction of the magnetic field H shifts towards a negative effect with decreasing film thickness and reaches zero at φ equal to 1450 and 1350 for films with a thickness of 600 Å.

On films with a thickness of 500 Å the magnitude of the magnetoresistance in the entire range of angle variation has a negative sign of the magnitude of the effect.

With a decrease in temperature following the transition of the Ni/MgO film to the “strongly magnetic” state a structural phase transition occurs in it.

Keywords: magnetoresistance, magnetoresistive element, singlecrystal films, transverse effect, magnetic field orientation, film planes, relative magnetization.

Introduction. Thin films and structures based on them have a number of unique physical and chemical properties not found in bulk materials [1–7], which makes them relevant the object of experimental and theoretical research for more than a decade.

A special place in these studies is occupied by films of ferromagnetic 3 – d metals (FM) iron (Fe), nickel (Ni) and cobalt (Co) - due to the possibility of their use in devices of solid-state

electronics (devices for processing and storing information, band-stop filters and phase shifters), as growth catalysts carbon nanotubes, orienting coatings for the growth of graphene films, films of various metals, dielectrics and semiconductors, promising for use in devices of solid-state micro- and nanoelectronics on the principles magnonics and spintronics. Along with this, ferromagnetic films and multilayer structures on their basis, they provide qualitatively new opportunities for optimization and miniaturization of the component base of solid-state microelectronics and development of sensors magnetic field, microwave current generators, spin transistors and other solid-state devices based on spin transport.

However, each of the above potential areas of application of thin ferromagnetic films imposes certain restrictions on the properties that they must have.

According to the structure of the crystal structure, films can be divided into 4 classes: amorphous films, polycrystalline films, textured films (i.e. polycrystalline films with a predominant crystallographic grain orientation in the selected direction) and epitaxial films.

Films of magnetically ordered substances (Ni, Fe, Co and magnetic alloys) occupy a special place in the physics of films. This is due to the fact that they allow solving a number of fundamental problems for "two-dimensional magnetism" as well as the fact that they have a number of specific magnetic properties: a specific domain structure and the associated magnetic anisotropy, "ripple of magnetization", etc. [8, 9] Recently, the phenomenon of giant magnetoresistance (GMR) has been added to them that has attracted especially great attention to magnetic films and is now the subject of comprehensive research.

The study of the properties of magnetic films is of particular importance due to their wide use in modern microelectronics [10-12].

The choice of Ni films as objects of study was due to a number of reasons. First, Ni is an excellent candidate for magnetic properties versus thickness because has the lowest Curie point ($T_c = 631$ K) in the series of ferromagnetic metals (Fe, Co); over the entire range of thicknesses and temperatures, Ni, in contrast to Fe and Co, does not have polymorphic transitions, retaining the fcc lattice responsible for ferromagnetism. With an increase in the film thickness nickel demonstrates both the dependence of the T_c on the thickness and the transition from two-dimensional "Ising" magnets to three-dimensional "Heisenberg" ones [13, 14]. Second, Ni films, both in the bulk state and in the film state [15], are the simplest, sort of "model," for studying their properties; moreover, to date, the electrical and, in particular, the galvanomagnetic properties of films have been little studied.

The effect of the substrate and the dependence on the angle φ between the [100] axis of single-crystal thin nickel films on the change in the electrical resistance of the films under the influence of a magnetic field or magnetization (magnetoresistance) has not been studied by anyone.

Recently [9] reported on the technology of growing thick monocrystalline films by chemical transport on a magnesium oxide substrate. The method makes it possible to obtain films with a thickness of $2 \mu\text{m} \leq d \leq 15 \mu\text{m}$, and they are characterized by a relatively high ratio of residual resistance $\eta = \rho_{300\text{K}}/\rho_{4,2\text{K}}$, reaching a value of 10^3 .

In accordance with the technology the films were grown at a substrate temperature of 1000°C . The thermal expansion coefficient of nickel, α_{Ni} , exceeds the corresponding coefficient α_{MgO} for magnesium oxide. For example, at room temperature $\alpha_{\text{Ni}} = 12,5 \cdot 10^{-6}$, $\alpha_{\text{MgO}} = 11,2 \cdot 10^{-6} \text{ deg}^{-1}$. Consequently, upon cooling, the film, being bonded to the substrate, is subjected to in-plane tensile stress, which leads to tetragonal deformation of the crystal lattice. This conclusion was confirmed by comparing the crystal lattice parameters measured by X-ray diffraction at room temperature T_k and $T = 77$ K.

The aim of this work is to study the magnetoresistance and angular dependence as well as the effect of the substrate and the dependence on the angle φ between the [100] axis on thin single-crystal films 500-600 Å thick at 295 K in magnetic fields up to 21 kOe.

Methods for measuring magnetoresistance. The magnetoresistance was measured by an unbalanced double Thomson bridge with an accuracy of the order of 10^{-6} Ohm. The magnetoresistance measurements were carried out in the longitudinal and transverse directions of

the external magnetic field, as well as depending on the angle φ (between the [100] axis and the direction of the external field). In all measurements the current passed through the samples remained constant 5×10^{-4} A. When measuring the longitudinal and transverse magnetoresistance the external magnetic field varied from 0 to 21000 Oe. The effect measurements in our experiments were carried out with an accuracy of 2-4 % on average.

When the magnetoresistance was measured, depending on the angle φ , the saturation field was equal to 5800 Oe. The directions of measurements of r , electric current i , external magnetic field H with respect to the [100] axis had the following orientations: for the transverse effect, when $[100] \parallel r \perp H$; for longitudinal $[100] \parallel i \parallel r \parallel H$.

The measurement of the magnetoresistance as a function of the angle φ was carried out at $[100] \parallel i \parallel r \wedge H = \varphi$.

The experimental part was carried out on thin single-crystal films obtained in vacuum (10^{-4} mm Hg) at a substrate temperature of 520 K for MgO. The film thickness was 5 μm ; the film was obtained by the method of chemical transport reactions. The samples were annealed at temperatures of 300, 325, 350, 400, and 700° C in air. The deposition rate was maintained at 0.1 nm/s, and the thickness of the Ni films was measured by piezoelectric microweighing (quartz microbalance).

Thermal oxidation was carried out at a heating rate of 2° C /min. Once the desired maximum temperature was reached, it was maintained for 3 hours to allow oxidation and free diffusion of Ni atoms on the surface. The cooling rate was set at 2.5° C / min.

It was assumed that the change in $\Delta\rho/\rho$ with a change in the shape of the film is associated with the Hall potential difference [16]. The [001] plane of thin nickel films was checked in the electron diffraction pattern.

Lead wires were fastened with a special solder consisting of pure elements (57% Ga + 23% In + 20 % Sn parts by weight), having a low melting point and ensuring contact reliability [17].

Results and discussion. A possible oxidation mechanism of a nickel film is schematically shown in Figure. 1.

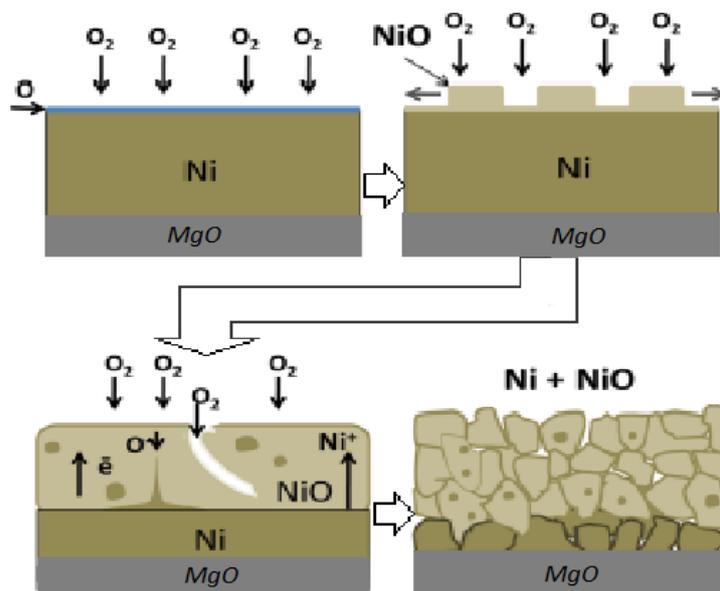


Figure 1. Schematic representation of the thermal oxidation process of a nickel film

Oxidation of nickel films occurs through the transfer of electrons, nickel cations and oxygen anions through the film. Annealing controls the oxidation process and film morphology.

Photographs of Ni films on MgO substrates after annealing at 300, 325, 350, 400, and 700° C are shown in Figure 2.

According to the results of the analysis of this study we find that Ni films 50 nm thick are almost completely oxidized at an annealing temperature of 700 ° C.

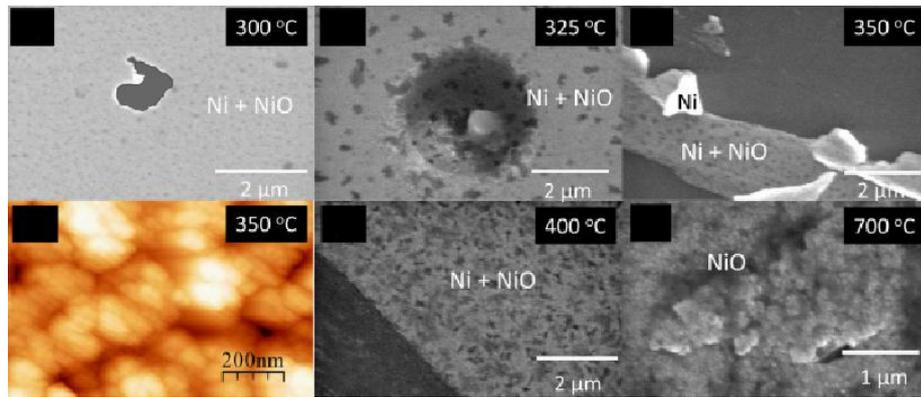


Figure 2 Micrographs of nickel films on Si/SiO₂ substrates at different annealing temperatures

The results obtained for the longitudinal and transverse magnetoresistance as well as the dependence of the magnetoresistance on the angle φ for single-crystal thin films of various thicknesses, are shown in Figure 3, 4 and 5. In figure 3 shows the graphs of the dependence $(\Delta\rho/\rho)$ on H of films obtained during the same technological cycle on a MgO substrate. The direction of the current during measurements was parallel to $[100]$, the direction H was perpendicular, the streamline was either in the plane of the film $(\Delta\rho/\rho)_{\perp}$, or along its normal $(\Delta\rho/\rho)_{\top}$.

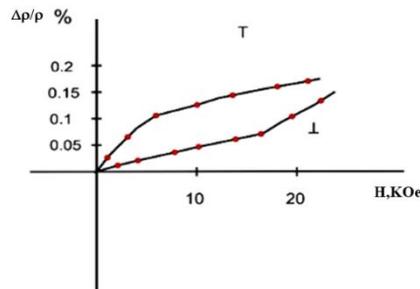


Figure 3. Graph of the field dependence $(\Delta\rho/\rho)_{\perp}$ and $(\Delta\rho/\rho)_{\top}$ of a nickel film on a MgO substrate. Film thickness 500 Å

Films on a MgO substrate experience thermoelastic stresses of different sign which is obviously associated with different dependences of their transverse magnetoresistance on the field H . For a Ni / MgO film in a field of 20 kOe $(\Delta\rho/\rho)_{\top}$ reaches a value of 0.17 %, and $(\Delta\rho/\rho)_{\perp}$ - values of 0.1 %.

It is assumed that tensile density stresses transform the Ni/MgO film into a “strong” (according to Campbell, see Figure 4) ferromagnet, characterized by a high magnitude of magnetoresistance (up to 0.25% at low temperatures). A well-known method of transferring nickel to the state of a “strong” ferromagnet is the introduction of impurities (Fe, Co, Cu, etc.) into it. The fact that large values of $\Delta\rho/\rho$ in Ni/MgO films are caused by mechanical stresses is evidenced by the fact that these values decrease with repeated heating-cooling cycles [18].

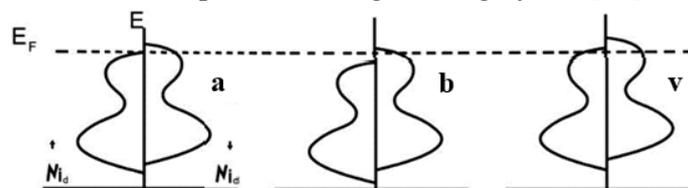


Figure 4. Diagrams of the density of states of the d band of nickel (a), “strong” (b) and “weak” (c) ferromagnets

When measuring the magnetoresistance, depending on the angle φ , a shift towards the negative effect is observed with decreasing film thickness (Figure 5).

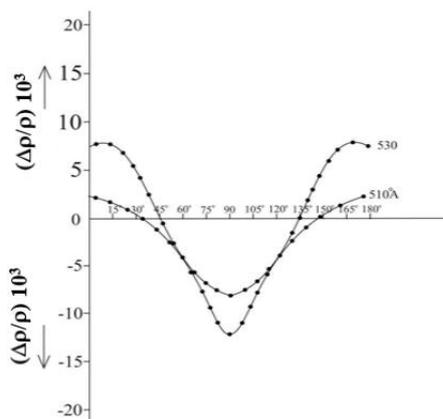


Figure 5. Dependence of the magnetoresistance $\Delta\rho/\rho$ on the angle $\varphi = [010] \parallel i \parallel r \wedge H$ in the field $H = 5800$ Oe

The method makes it possible to obtain films with a thickness of $2 \mu\text{m} \leq d \leq 15 \mu\text{m}$, and they are characterized by a relatively high ratio of residual resistance $\eta = \rho_{300\text{K}}/\rho_{4,2\text{K}}$, reaching a value of 10^3 .

In this work the anisotropy of the transverse magnetoresistance of single-crystal nickel films was studied. The measurements were carried out on samples whose surface plane coincided with the [001] plane. We used samples with a thickness of $d = 5.0 \pm 0.5 \mu\text{m}$ with a ratio $\eta = 10^3$.

Figure 6 shows the angular dependences of the transverse magnetoresistance $(\Delta\rho/\rho)(\varphi)$ of such films at $T = 4.2$ K and the direction of the current along [110]. Here $\Delta\rho = \rho_H - \rho$, where ρ_n, ρ are the resistivity of the sample in an external field H and in the absence of a field ($H = 0$). The angle φ determines the direction H relative to the normal to the film plane (at $\varphi = 90^\circ H \parallel n$). Curves 1, 2 show the dependences $(\Delta\rho/\rho)(\varphi)$ for a film connected to a substrate and, therefore, under the action of tensile stresses, at $H = 14$ (1) and 21 kOe (2). Note that repeated cooling and heating of the film on the substrate from T_c to $T = 4.2$ K did not change the $(\Delta\rho/\rho)(\varphi)$ dependence. Curve 3 is a graph of $(\Delta\rho/\rho)(\varphi)$ at $H = 21$ kOe on the same film, but separated from the MgO substrate.

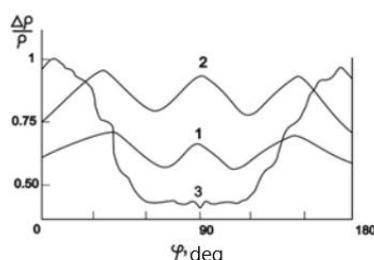


Figure 6. Angular dependences of the transverse magnetoresistance $(\Delta\rho/\rho)(\varphi)$ of films at $T=4.2$ K and the direction of the current along [110]

After removing the film from the substrate the ratio decreased to ~ 870 . As can be seen from a comparison of curves 2 and 3 stress relief leads to a noticeable change in the angular dependence of the transverse magnetoresistance of nickel films. It should be noted that at $T = T_c$ and $T = 77$ K the curves $(\Delta\rho/\rho)(\varphi)$ obtained by us coincide with the known (3) results for the anisotropy of the magnetoresistance of bulk samples. Moreover, as it turned out, the separation of the film from the substrate does not change the dependence $(\Delta\rho/\rho)(\varphi)$ at the indicated temperature values.

The angular dependence of the magnetoresistance of the film removed from the substrate at $T=4.2$ K (Figure 6 (3)) generally corresponds to the graph $(\Delta\rho/\rho)(\varphi)$ of the transverse magnetoresistance of a single-crystal nickel rod with $\eta = 2700$ [11]. In this work the anisotropy of the magnetoresistance was associated with the shape of the Fermi surface of nickel. In contrast to

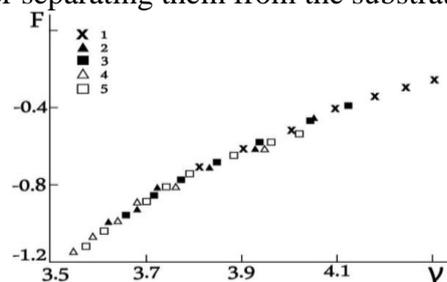
[9], the curve obtained by us does not have sharp minima for the H directions coinciding with the crystallographic axes of the [111] type and the minima for H [100] do not reach the value noted by the authors [11]. Such differences, in our opinion, are associated with the scattering of electrons at the boundaries of the sample. In the magnetic field used to measure the magnetoresistance of nickel films the cyclotron radius of conduction electrons is $r \geq d$. Note that our data are closer to the curve $(\Delta\rho/\rho)(\varphi)$ given in the work of Fossett and Reed than the results of [12] where measurements were carried out on a massive sample with $\eta = 1000$. As is known, the static resistance of films in a magnetic the field is determined by the electron mean free path l_{eff} into which the carrier scattering on the film surface contributes. At the same time, the samples contain electrons that do not collide with the surface and have a mean free path $l > l_{\text{eff}}$. Under the condition $l \gg r$, such electrons can contribute to the film magnetoresistance determined by the shape of the Fermi surface, that is observed experimentally.

Thus, the study of the anisotropy of the magnetoresistance of a nickel film on an MgO substrate makes it possible to conclude that the shape of the Fermi surface of conduction electrons is changed due to the tetragonal deformation of the crystal lattice of the deposited material. As far as we know, such significant changes in $\Delta\rho/\rho$ under the action of external stresses in nickel have not been observed previously [19].

It is noteworthy that the value of $\Delta\rho/\rho$ in the film on the substrate at $H \parallel n$ ($\varphi = 90^\circ$) is greater than at H in the plane of the sample, despite the lower value of the induction B in the first case. Indeed, in a ferromagnetic film sample, magnetized to saturation, at $H \parallel n$, the demagnetizing field is $H_{\text{rasm}} = -4\pi M$, as a result, $B \approx H$. At H in the film plane, $B = H + 4\pi M$. It is obvious that a decrease in induction cannot explain the minimum in the dependence $(\Delta\rho/\rho)(\varphi)$ on a film removed from a substrate at $\varphi = 90^\circ$.

When the current is directed along [100] (in this case, when H is in the film plane and when $H \parallel n$, the magnetic field is applied along equivalent crystallographic directions) the study of the dependence of $\Delta\rho/\rho$ on H showed that in fields greater than the saturation field the same values of $\Delta\rho/\rho$ for the above-mentioned mutually perpendicular directions of the field we obtain for H differing by 6 kOe. This is approximately equal to $4\pi M$ in nickel. The dependence of $\Delta\rho/\rho$ on the magnitude of the magnetic field in the region $H < 21$ kOe for any φ is a monotonically increasing function of H. kE. The exit of M in the direction n is facilitated by the induced perpendicular anisotropy caused by the action of tensile stresses on the film.

As is known, in a number of cases, for example, when plotting the Kolerov curves [7] it is required to know the resistance of a ferromagnet at $B = 0$. Due to the fact noted above that at $M \parallel n$ the magnetic induction of the film is $B \approx H$ the value of its resistance at $B = 0$ is by a simple extrapolation of the dependence $\rho(H)$ obtained in the region of magnetic saturation of the sample at $H \parallel n$ to the value at $H = 0$. Using this procedure we obtained the dependences $(\delta\rho/\rho_0)(H)$ at H in the film plane and the measuring current directed along [100]. Here $\delta\rho = \rho_B - \rho_0$, where ρ_B - the value of the resistance of the sample with an induction field in it equal to B; ρ_0 is the resistance at $B = 0$. The measurements were carried out on three samples on an MgO substrate differing in the value of η and in thickness. Two samples with $d = 9 \mu\text{m}$ had $\eta = 800$ (sample No. 1) and $\eta = 460$ (sample No. 2), respectively. Sample no. 3 with $d = 5 \mu\text{m}$ had $\eta = 380$. Measurements $(\delta\rho/\rho_0)(H)$ on samples no. 2, 3 were repeated after separating them from the substrate.



**Figure 7. Dependences of $F = \log(\delta\rho/\rho)$ on $\gamma = \log(Bnd^*/d)$ of single-crystal nickel films
1-3 - films on a substrate; 4, 5 - films separated from the substrate
 d (μm), η : 1 - 9.800; 2 - 380; 3 - 9.460; 4 - 5.250; 5 - 9.380**

Figure 7 shows the universal dependence, $\delta\rho/\rho = f(B\eta d^*/d)$ which obeys the magnetoresistance of the studied samples; here $d^* = 9 \mu\text{m}$. The possibility of representing the magnetoresistance as a universal curve depending on the thickness of the sample indicates the presence of a size effect the existence of which was assumed when discussing the results of studies of the anisotropy of magnetoresistance.

Thus, we have demonstrated the effect of tensile stresses acting on it from the side of magnesium oxide on the transverse magnetoresistance of a single-crystal nickel film.

Conclusions. The modification of the anisotropy of the magnetoresistance of a film on a substrate as compared to a free sample is apparently associated with a change in the shape of the Fermi surface of carriers. In this case, the volume occupied by carriers in momentum space does not change that is indicated by the existence of a general universal dependence for the magnetoresistance of films on a substrate and films separated from it.

The magnetoresistance in both longitudinal and transverse magnetic fields for all investigated film thicknesses has a different course of curves: in the first case- with a positive value, in the second-with a negative value of the effect magnitude.

The magnitude of the magnetoresistance at a saturation field of 5800 Oe depending on the angle φ between the [100] axis and the direction of the magnetic field H shifts towards a negative effect with decreasing film thickness and reaches zero at φ equal to 1450 and 1350 for films with a thickness of 600 Å.

On films with a thickness of 500 Å the magnitude of the magnetoresistance in the entire range of angle variation has a negative sign of the magnitude of the effect.

With a decrease in temperature following the transition of the Ni/MgO film to the “strongly magnetic” state a structural phase transition occurs in it.

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OPTICAL SYSTEM AND FLOW CELL FOR STUDYING THE POLARIZATION SPECTRA OF SELF-ASSEMBLED MOLECULES

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Abstract:

Background. The authors developed, patented and applied a flow-through optical cuvette that allows recording the polarization spectra of self-aggregates of vitamins and food dyes. Equipment polarimeters and dyregrov flow-through cuvettes allows you to take spectra of linear or circular dichroism. These spectra allow us to determine the fine details of optical transitions. And also detect the hiding of a band of self-aggregated molecules, as in the case of γ -formylmesoporphyrin, Riboflavin. They allow you to get information of a diverse nature. The structure of self-assembled molecules is needle-like and they have optical activity in the hydrodynamic flow.

The article reveals the relevance of the research and justifies the purpose of the work which is to develop optical systems and cuvettes that allow increasing the sensitivity of optical devices as a result of which conditions are manifested to register the weak polarization characteristic of molecules.

Methods. The experimental method shows an optical system in the form of a double Fresnel parallelepiped that increases the sensitivity of the optical device by two orders of magnitude and also shows the optical devices used to remove the absorption and fluorescence spectrum. The use of solvents in the study of vitamin preparations and certain food dyes is justified.

Results. The principle of operation that allows an optical cell of constant thickness to register linear dichroism of weakly oriented molecules in a hydrodynamic flow is given. This section also presents the spectra of negative rotation of the polarization plane of the associates of γ -formylmesoporphyrin and Riboflavin in a laminar hydrodynamic flow.

Results and discussion the results of studies on self-aggregation of vitamin B2 in a binary mixture of water+dioxane solvents are presented. To remove the linear dichroism spectra of self-aggregated molecules, γ -formylmesoporphyrin, and vitamin B2, a constant-thickness flow cell was used.

Conclusion. Thus, it is shown that polarimeters and dichrographs are equipped with a double Fresnel parallelepiped that is installed in front of the pokkels cell with a KDP (KN2 PO4) crystal and allow recording the spectrum of linear dichroism of weakly oriented molecules of food pigments and vitamins. In this case, the sensitivity of the device increases by about two orders of magnitude. The developed optical system is used to measure the linear dichroism spectra on the polarimeter spectrum when the films are oriented with the axis of its extension at an angle of 40° as the polarization vectors of the incident light. It is shown that the structure of self-assembled molecules is needle-like and they have optical activity in a hydrodynamic flow.