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MAGNETISM
AND FERROELECTRICITY

Synthesis and Study of the Magnetic Characteristics of Nanocrystalline Cobalt Films

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Abstract—The effect of various technological factors on the main magnetic characteristics of nanocrystalline cobalt films obtained by chemical deposition is studied. The effective saturation magnetization, uniaxial anisotropy, coercive force, and other characteristics of local spots of films were measured using a scanning FMR spectrometer over the decimeter wavelength range. A technique for preparing substrates is developed, and the optimum technological conditions of thin-film synthesis are found making it possible to significantly decrease magnetic inhomogeneities across the area and thickness of samples. It is shown that one can significantly decrease the FMR linewidth and increase the magnetic permeability of films in a given microwave frequency range.

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1. INTRODUCTION

Thin films of magnetically soft materials find wide application in recording/reading heads, sensors of weak magnetic fields, and various controlling devices operating in the microwave (mw) frequency range, such as filters, amplitude modulators, power limiters, and phase changers. For these devices, one of the most important parameters of films is the high magnetic permeability μ in combination with low mw power losses in a given frequency range. Therefore, a search for new physical principles and new technology for producing high-μ films and improvement of existing techniques are important problems in the modern physics of magnetic phenomena. In order to solve these problems, one should, above all, develop methods for increasing the saturation magnetization M_0 of materials and decreasing the ferromagnetic resonance (FMR) linewidth ΔH, which (in thin films) is determined, to a great extent, by the degree of inhomogeneity of the magnetic characteristics across the aria and thickness of samples.

It is well known that amorphous metallic films have a relatively high mw magnetic permeability. In comparison with polycrystalline samples, such films have narrower FMR lines due to the absence in them of the mechanism of line broadening associated with a spread in the orientations of the magnetocrystalline anisotropy axes (in contrast to usual polycrystals, where such spread exists due to random orientations of crystallites). There are various techniques developed for producing such amorphous films. However, the chemical deposition technique [1, 2] (employed in this work) is distinguished by its simplicity, accessibility, and cheapness.

It should be stressed that the properties of samples prepared through chemical synthesis depend strongly on a variety of technological factors, such as the composition and concentration of salts in a solution, the solution temperature, the material and quality of the substrate, and the technique employed for preliminary treatment of the substrate. Moreover, the film parameters depend substantially on the characteristics of the external magnetic field (which is usually applied during film deposition), such as its magnitude and uniformity and its orientation with respect to the substrate axes. Therefore, in order to determine the optimum conditions for producing films with extremely high values of certain characteristics, one should usually perform a comprehensive technological study. In particular, we performed studies with the aim of obtaining the minimum possible FMR linewidth in cobalt films in combination with the maximum value of the effective saturation magnetization. We also developed a technique that makes it possible to decrease magnetic inhomogeneities across the area and thickness of samples and thereby obtain extremely high values of the mw magnetic permeability.

2. EXPERIMENTAL

It is well known that chemically deposited films of cobalt, nickel, iron, and their alloys doped with phos-

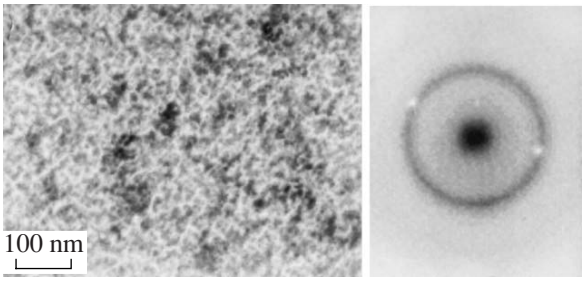

Fig. 1. Microphotograph of a spot of a chemically deposited film and its electron diffraction pattern.

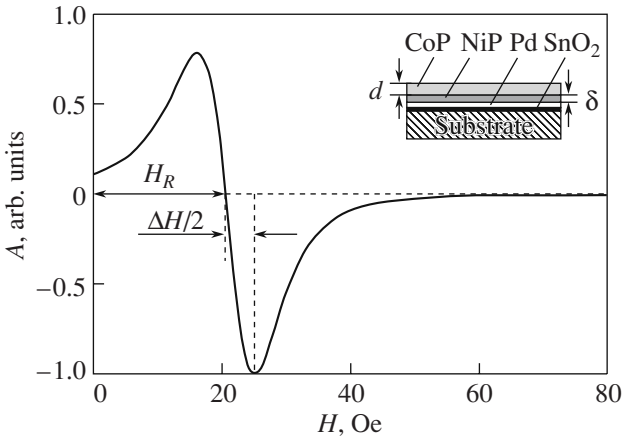

Fig. 2. Sample under study (schematic) and a differential curve of the FMR line recorded for a local spot of a 10-nm-thick cobalt film using a scanning FMR spectrometer.

phorus to a fairly low concentration (which favors the formation of a structure that is amorphous in terms of x-ray diffraction) have a high saturation magnetization and a relatively narrow FMR linewidth; i.e., they can possess a fairly high mw magnetic permeability. The technology for deposing such films on dielectric substrates requires preliminary activation of the substrate surface by a palladium sublayer, which has been found to have a polycrystalline structure. As a magnetic film is deposited on this sublayer, its growth is epitaxial in the initial stage. As a result, the first layers of the magnetic film are not amorphous but rather repeat the structure of the palladium sublayer. As the film grows further, it gradually becomes x-ray amorphous due to the presence of a small amount of phosphorus in its composition; the phosphorus is precipitated during synthesis and thereby favors amorphization.

Figure 1 shows a typical photograph of a spot of a chemically deposited 60-nm-thick x-ray amorphous film and its electron diffraction pattern obtained using a transmission electron microscope. The strong first and weak second diffraction maxima observed against the background of a diffuse halo indicate that there is a short-range order. Thus, in actuality, the structure of an x-ray amorphous film is not amorphous; the reflections in the electron diffraction pattern correspond to interplanar spacings $\lambda_1 \approx 2.03$ Å and $\lambda_2 \approx 1.22$ Å. Such films usually have a nanocrystalline structure with crystallites 2 to 6 nm in size separated by thin phosphorus interlayers. For this reason, chemically deposited x-ray amorphous films are called nanograined.

Usually, films are deposited in the presence of a uniform dc magnetic field applied in the substrate plane in order to decrease magnetic inhomogeneities across the sample area and make the FMR lines narrower. However, as the film thickness decreases starting from approximately 50 nm, the FMR linewidth increases rapidly by at least one order of magnitude [3] even if the films are deposited in the presence of a relatively strong uniform magnetic field. This is due to the fact that, as indicated above, a film grows epitaxially in the initial stage and its atoms reconstruct the polycrystalline structure of the palladium sublayer. As a result, nanograins in the first layers of the film are oriented almost randomly, which leads to a significantly broadened FMR line.

It should be stressed that the dc magnetic field applied during film synthesis produces uniaxial in-plane magnetic anisotropy in the film due mainly to magnetostriction and elastic stresses arising in the interface between the film and substrate [4]. It is interesting that, in weak fields, the induced uniaxial anisotropy increases in proportion to the applied field and then saturation is reached in fields of ~1.5 Oe [5]. In order to obtain an isotropic film, the parasitic magnetic fields should be balanced with an accuracy of better than 10^{-3} Oe [6].

As shown in [7], the high-frequency properties of chemically deposited x-ray amorphous magnetic films are significantly improved if, prior to the deposition, the palladium sublayer is preliminarily covered with a nonmagnetic buffer amorphous layer of a certain thickness. This buffer layer can be a chemically deposited Ni–P film containing phosphorus in greater than a 7% concentration, which makes the buffer layer nonmagnetic [8]. We studied the main magnetic characteristics of such multilayer structures and the effect on them of various technological factors using nanocrystalline Co films with about 4% P deposited on 2.5-mm-thick glass substrates 12 × 12 mm in lateral size. The substrates were cut with a diamond cutter from thoroughly washed standard photoplates designed to produce masks for microelectronic photolithography.

A buffer nonmagnetic amorphous Ni–P layer of thickness δ was deposited at 90°C on substrates which were preliminarily treated by a technique developed in [9] including the deposition of a tin oxide layer a few atomic layers thick (see inset to Fig. 2) and a 5-nm-

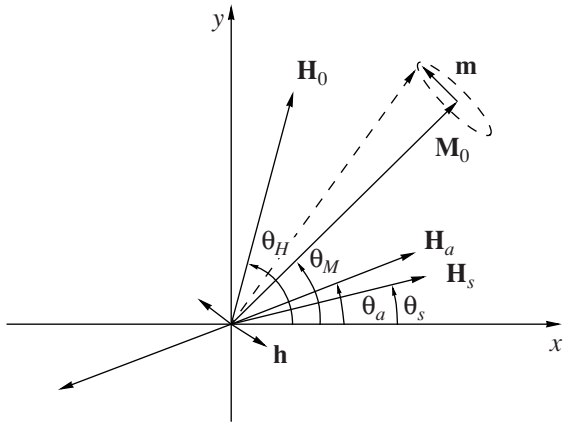

Fig. 3. Geometry of a magnetic film with in-plane uniaxial and unidirectional anisotropy.

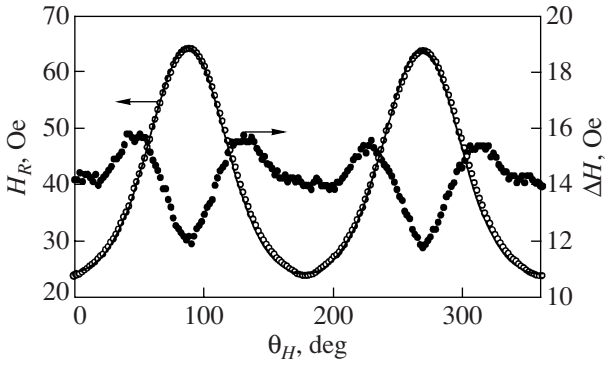

Fig. 4. Angular dependences of the resonance field H_R and the FMR linewidth ΔH: points are experiment and the solid line is calculation.

thick palladium layer. A Ni–P film was deposited from a solution with the following composition: nickel sulfate (7 g/l), sodium hypophosphite (10 g/l), sodium citrate (25 g/l), salmiac (17 g/l), and ammonia (0.7 ml/l); the hydrogen ion exponent of the solution pH = 8.5.

The multilayer structures thus obtained were used as a substrate to deposit nanocrystalline Co–P films with thickness d at 97°C in a uniform in-plane dc magnetic field H = 3 kOe directed along a film side. The films were deposited from a solution with the following composition: cobalt sulfate (30 g/l), sodium hypophosphite (50 g/l), sodium citrate (80 g/l), and ammonia (3 ml/l); pH = 9.0. The layer thicknesses were determined from the deposition time. For this purpose, the growth rates were preliminarily measured for each layer using an x-ray reflectometer, which permits one to determine film thicknesses with an accuracy of better than ±1 nm.

The magnetic parameters of local spots of the samples thus obtained were measured using an automated scanning FMR spectrometer [10], with a sensor being a microstrip resonator (MSR) prepared on a substrate with a high permittivity. Near an antinode of a mw magnetic field, the resonator ground plane has a small hole for making local measurements. Resonance mw power absorption of a spot of the film sample in the FMR spectrometer is detected in the usual fashion (using the modulation technique [11]) by measuring the Q factor of the MSR over the course of scanning of a static magnetic field applied in the film plane. The resonator is a driving circuit in a transistor mw generator located, together with a detector, within the sensor case. The measuring hole in the MSR is used simultaneously as a localized source of a mw magnetic field and as a coupling channel between the film under study and the resonator.

The main advantage of the scanning spectrometer is its high sensitivity owing to the fact that the sample occupies most of the sensor volume due to the smallness of the MSR and to the use of low-frequency (1 kHz) modulation of the scanning static magnetic field with subsequent locked-in detection of the signal. An advantage is also that the spectrometer is equipped with a set of mw sensors covering a fairly wide frequency range (0.1–6.0 GHz), which is important in studying magnetic inhomogeneities of thin films. Indeed, it is well known that certain characteristics of magnetic films, such as magnetostriction and bulk and surface magnetic anisotropies, are dependent on the magnitude of the static magnetic field H applied to the sample. Moreover, inhomogeneities of the distributions of these quantities over the film plane, as a rule, becomes less pronounced as H increases. Since the FMR field increases almost linearly with the pump frequency f, one should significantly decrease the frequency f in order to measure the profile of magnetic inhomogeneities of films in weak fields. However, in standard spectrometers with pump frequencies of the order of 10 GHz, the resonance magnetic fields are very high even when the field is applied in the film plane and, hence, the demagnetization factors are close to zero. For permalloy films, e.g., the resonance fields are ~10^3 Oe.

In this work, we employed a scanning spectrometer with a mw sensor operating at a pump frequency f = 2.274 GHz and the resonance fields did not exceed 75 Oe for the samples under study. The diameter of the measuring hole of the sensor was ~1 mm; therefore, local measurements were performed for a spot area of ~0.8 mm^2. The angular dependences of the resonance field H_R and the linewidth ΔH were measured for each local spot of the film surface in steps of 1 mm. From these dependences, the main magnetic characteristics of the films were determined automatically with a computer program based on a phenomenological calculation [12]. In contrast to [12], the calculation in this

work includes the unidirectional anisotropy [13], although it is small in comparison with the uniaxial anisotropy in the films under study. The scanning FMR spectrometer was also used to measure the local values of the coercive force; for these measurements, magnetization reversal was performed along the easy axis [13].

Based on the angular dependence of H_R, we calculated the saturation magnetization, the magnitude and orientation of the uniaxial and unidirectional magnetic anisotropy fields, and the minimum FMR linewidth. The FMR spectra were recorded in the regime of reverse magnetic-field sweeping in order to eliminate the effect of hysteretic phenomena in films on the FMR spectrum. For this purpose, the film was also magnetized by a field at least an order of magnitude higher than the uniaxial anisotropy field prior to recording the FMR spectrum after each rotation of the sample through a certain angle. The half-width of the FMR line is defined as the difference between the field corresponding to the extremum in the differential absorption curve located on the right of the resonance field (Fig. 2) and the resonance field. This definition permits us to eliminate the ambiguity in determining ΔH caused by the asymmetric shape of the FMR line, which, in turn, is due to the distortion of the left-hand slope of the resonance curve caused, e.g., by the nonresonant mw susceptibility of the anisotropic film [14].

3. RESULTS OF THE STUDIES

Let us consider the case where a thin magnetic film lies in the *xy* plane (Fig. 3) and the external static magnetic field $\mathbf{H}_0$ and the mw magnetic field $\mathbf{h}$ (which varies according to a harmonic law with a circular frequency ω) are directed in the film plane at angles θ_H and $\theta_H + \pi/2$ to the *x* axis, respectively. The angles that the uniaxial magnetic anisotropy field $\mathbf{H}_a$ (directed along the easy axis) and the unidirectional anisotropy field $\mathbf{H}_s$ make with the *x* axis are θ_a and θ_s, respectively.

The free energy density *F* of the film is the sum of the densities of the Zeeman energy (F_H), the uniaxial (F_a) and unidirectional (F_s) anisotropy energy, and the energy of magnetic charges arising on the film surfaces due to magnetization precession (F_M):

$$\begin{aligned} F &= F_H + F_a + F_s + F_M \\ &= -(\mathbf{MH}) - \frac{H_a}{2M_0}(\mathbf{Mn})^2 - (\mathbf{MH}_s) + \frac{1}{2}(\mathbf{M}N\mathbf{M}). \end{aligned} \quad (1)$$

Here, $\mathbf{H} = (\mathbf{H}_0 + \mathbf{h})$; $\mathbf{M} = (\mathbf{M}_0 + \mathbf{m})$, where $\mathbf{M}_0$ and $\mathbf{m}$ are the static and dynamic components of the magnetic moment $\mathbf{M}$, respectively; $\mathbf{n}$ is a unit vector along the easy axis; and *N* is the demagnetization factor tensor,

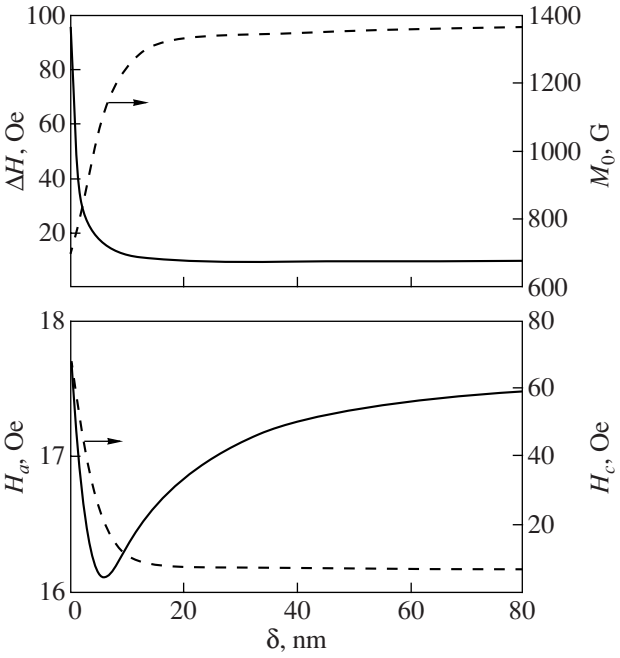

Fig. 5. Dependences of the FMR linewidth ΔH, the effective saturation magnetization M_0, the anisotropy field H_a, and the coercive force H_c of a cobalt film on the buffer layer thickness.

which has only one nonzero component ($N_{zz} = 4\pi$) in the case of a thin film.

The motion of the magnetic moment $\mathbf{M}$ under a magnetic field is described by the Landau–Lifshitz equation

$$\frac{\partial \mathbf{M}}{\partial t} = -\gamma[\mathbf{M} \times \mathbf{H}_{\text{eff}}], \quad (2)$$

where γ is the gyromagnetic ratio and $\mathbf{H}_{\text{eff}} = -\partial F/\partial \mathbf{M}$.

Solving Eq. (2) in a linear approximation under the assumption that $m \ll M_0$ and $h \ll H_0$, it is easy to obtain the following relation between the resonance magnetic field H_R and the magnetic characteristics of the sample at a fixed pump frequency ω:

$$\begin{aligned} \left(\frac{\omega}{\gamma}\right)^2 &= [H_R\cos(\theta_H - \theta_M) + H_a\cos 2(\theta_a - \theta_M) \\ &+ H_s\cos(\theta_s - \theta_M)][4\pi M_0 + H_R\cos(\theta_H - \theta_M) \\ &+ H_a\cos^2(\theta_a - \theta_M) + H_s\cos(\theta_s - \theta_M)], \end{aligned} \quad (3)$$

where the angle θ_M defines the equilibrium direction of the saturation magnetization $\mathbf{M}_0$ of the film and can be found from the equation

$$\begin{aligned} H_0\sin(\theta_H - \theta_M) + \frac{1}{2}H_a\sin 2(\theta_a - \theta_M) \\ + H_s\sin(\theta_s - \theta_M) = 0, \end{aligned} \quad (4)$$

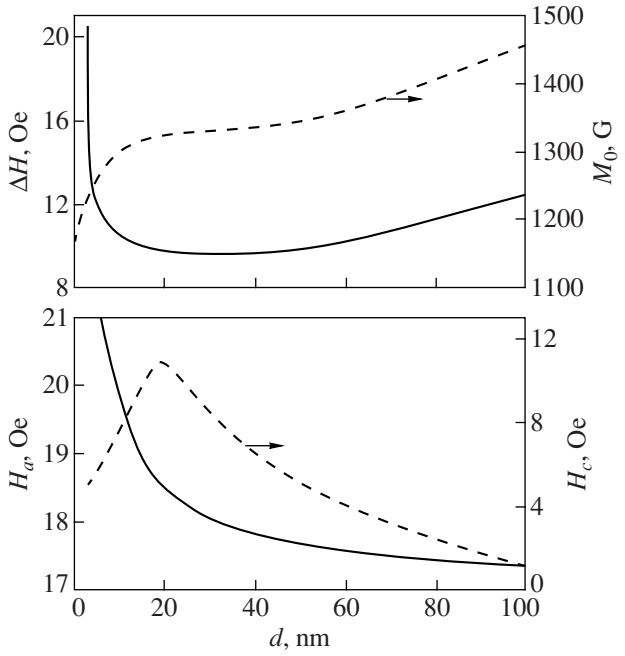

Fig. 6. Dependences of the FMR linewidth ΔH, the effective saturation magnetization M_0, the anisotropy field H_a, and the coercive force H_c of a cobalt film on the film thickness.

which can be derived by minimizing the free energy density F in Eq. (1).

In general, M_0 is the effective saturation magnetization [15], because our simple model does not take into account other factors influencing the magnitude of M_0 (such as elastic stresses and the perpendicular anisotropy caused by the film growth and the stresses).

Given the pump frequency ω, Eqs. (3) and (4) make it possible to determine the magnetic characteristics of any local spot of the film from the measured angular dependences of the resonance field $H_R(\theta_H)$ [13].

Figure 4 shows the resonance field H_R (points) measured for various values of the angle θ_H of the magnetic field in steps of 2° for a spot of a 10-nm-thick film. The solid curve is the least-squares fit of the theoretical angular dependence of H_R to the experimental points for the following parameter values of the film spot: $M_0 \approx 1270$ G, $H_a \approx 19.8$ Oe, $\theta_a = -4°$, $H_s \approx 0.85$ Oe, and $\theta_s = -158°$. As already mentioned, these characteristics were determined automatically from the measured $H_R(\theta_H)$ values. In essence, in order to determine the magnetic parameters, a polar coordinate frame was reduced to the center of symmetry of the experimental $H_R(\theta_H)$ curve also constructed in a polar coordinate frame [12].

Measured parameters averaged over the sample area and the standard deviations of these parameters

Sample	Averaged parameter value / Standard deviation				
	M_0, G	H_a, Oe	θ_a, deg	H_s, Oe	ΔH, Oe
a	1305 / 216	14.19 / 11.17	–53 / 180	0.24 / 3.05	11.49 / 8.12
b	1282 / 94	12.07 / 12.74	–3.18 / 180	0.29 / 1.03	12.63 / 4.53
c	1365 / 47	17.35 / 1.19	0.57 / 5.50	0.06 / 0.21	10.28 / 2.17

The accuracy with which the magnetic characteristics of thin magnetic films are determined is dependent, above all, on the accuracy in determining the resonance fields, which, in turn, depends on the intensity and width of the FMR line of a specific sample. For the films under study, the field H_R was measured to within ±0.02 Oe. However, since the angular dependences obtained are based on the large number of experimental points, the accuracy in determining the magnetic characteristics of the samples is significantly better due to averaging over these points. In this case, obviously, the measurements require a long time. However, it was established experimentally in [10] that, for the film characteristics to be determined with the required accuracy, it is sufficient to perform measurements of the angular dependences of the resonance field at five or six points in steps of 5°–10° near each extremum.

It should be noted that, when comparing the FMR linewidths ΔH of different samples, we chose the minimum values of the angle-dependent width $\Delta H(\theta_H)$, which, as a rule, correspond to the case where the magnetic field is perpendicular to the easy axis. The significantly increased FMR linewidths for other orientations of the magnetic field (Fig. 4) are caused, above all, by the deviation of the equilibrium direction of the magnetic moment from the direction of the magnetic field due to the film anisotropy (see Eq. (4)).

For one series of experiments, we synthesized (using the technique described above) 28 nanocrystalline magnetic Co–P films differing only in terms of the thickness of the nonmagnetic buffer Ni–P layer, $\delta = 0$–80 nm. The thickness of the main magnetic layer was the same for all of the samples, $d = 35$ nm. We measured the magnetic characteristics of a spot ~0.8 mm^2 in area located in the center of each sample. Figure 5 shows the dependences of some of these characteristics on the thickness of the nonmagnetic buffer layer. We note that, in the absence of a buffer layer ($\delta = 0$), the FMR linewidth of the film is maximum ($\Delta H \approx 90$ Oe) and the FMR signal is at least one order of magnitude smaller. The other magnetic parameters also have extremum values: the effective saturation magnetization is

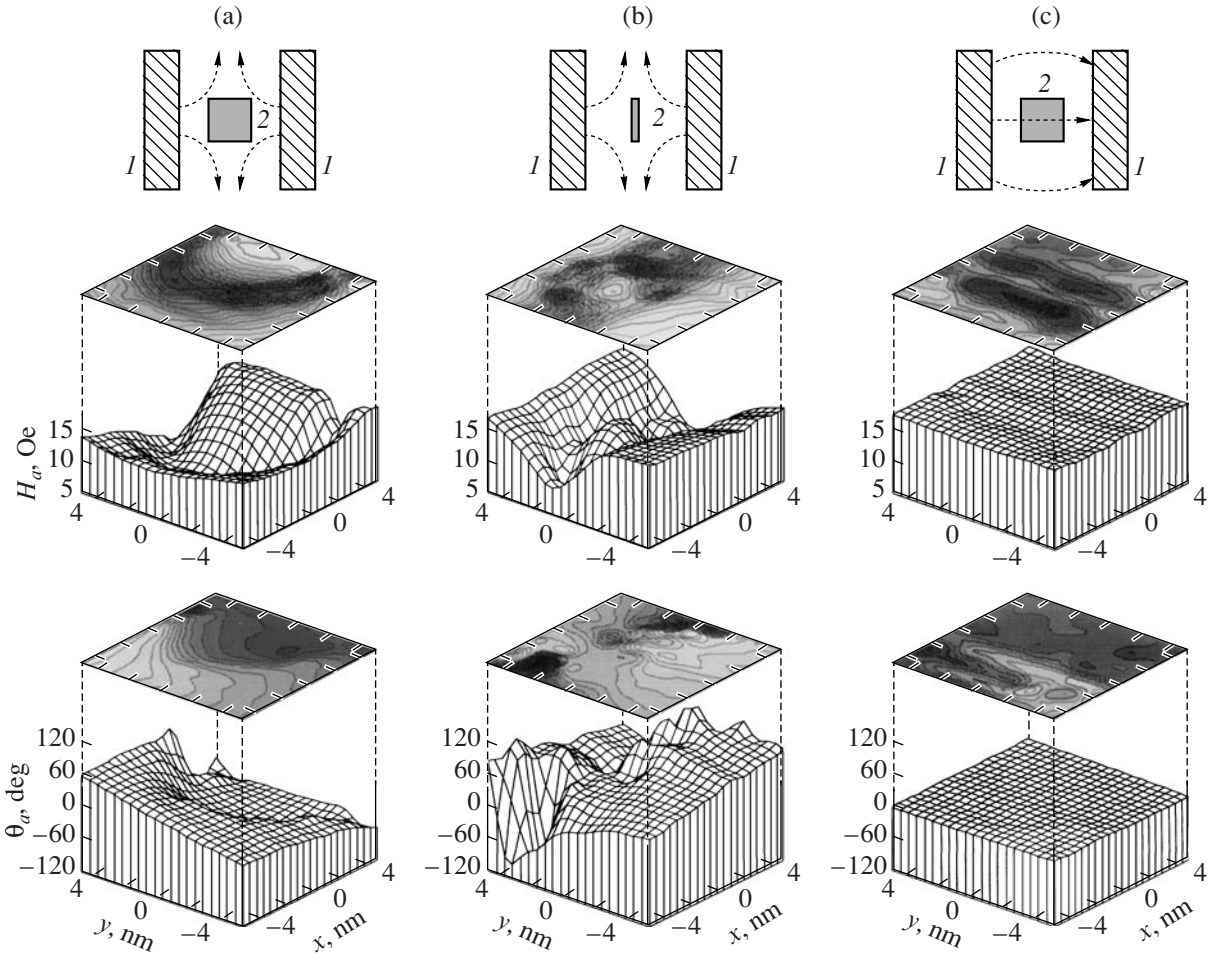

Fig. 7. Spatial distributions of the magnitude and orientation of the uniaxial magnetic anisotropy field of (a, b) films deposited in a gradient magnetic field and (c) a reference film deposited in a uniform magnetic field. Deposition geometry: (*1*) magnets and (*2*) substrate.

the smallest ($M_0 \approx 700$ G) and the coercive force and the unidirectional anisotropy field reach their maximum values ($H_c \approx 70$ Oe, $H_s \approx 6$ Oe). This fact can be explained by structural changes in the first cobalt layers in the interface with the palladium sublayer (wherein the change from polycrystalline to x-ray amorphous structure occurs) and by large elastic stresses strongly inhomogeneous across the film thickness caused by these changes.

As the thickness of the nonmagnetic x-ray amorphous Ni–P layer increases, the structural changes in the interface with the palladium involve this layer and the elastic stresses decay gradually in it. As a result, elastic stresses are almost absent in the magnetic Co–P film deposited on this buffer layer and the film becomes homogeneous across its thickness, as indicated by the fact that the FMR linewidth, the coercive force, and the unidirectional magnetic anisotropy decrease about tenfold. In this case, the effective saturation magnetization increases about twofold. The uniaxial magnetic anisotropy field H_a varies only slightly (to within 10%), reaching a minimum at a buffer layer thickness $\delta \approx 5$–6 nm. It should be noted that all measured quantities change most greatly over the range $\delta \approx 0$–10 nm; at $\delta \approx 30$ nm, almost all dependences reach saturation. The easy axis in the cobalt film always coincides in direction with the dc magnetic field that was applied during film deposition.

For another series of experiments, we prepared 60 samples differing only in the thickness of the main magnetic Co–P layer, which was varied in small steps over the range $d = 1$–100 nm. The buffer layer thickness was the same in all samples ($\delta = 20$ nm). The angular dependences of the FMR spectra and magnetization curves (from which the magnetic characteristics of the films are calculated) were also measured for local spots ~0.8 mm^2 in area located in the center of the samples. The dependences of some of the measured parameters on the thickness of the magnetic film are shown in Fig. 6. As the thickness d increases, both the uniaxial

and unidirectional anisotropy fields and the FMR linewidth decrease sharply. However, the latter begins to increase monotonically at "large" film thicknesses due to the skin effect. The effective magnetization first increases sharply to $M_0 \approx 1330$ G and reaches saturation at $d \approx 20$ nm; then, starting from $d \approx 60$ nm, it increases monotonically. The dependence of the coercive force on the thickness of the magnetic film exhibits a pronounced maximum ($H_c \approx 11$ Oe) at $d \approx 20$ nm. It is likely that, at this film thickness, the domain walls change from the Néel to Bloch type [16].

For the sample having the thinnest magnetic layer in this series ($d = 1$ nm), the measured characteristics are as follows: $M_0 \approx 1180$ G, $H_c \approx 5.5$ Oe, $H_s \approx 1.7$ Oe, $H_a \approx 23$ Oe, and $\Delta H \approx 20$ Oe. We note that, for the samples without a buffer Ni–P layer (in which the Co–P layer was deposited immediately on the palladium sublayer), the minimum cobalt film thickness at which the FMR signal can be reliably separated from the background noise is approximately threefold greater. For this film, the measured parameter values are as follows: $M_0 \approx 690$ G, $H_a \approx 19$ Oe, $H_c \approx 77$ Oe, $H_s \approx 6.5$ Oe, and $\Delta H \approx 100$ Oe.

As indicated above, the FMR linewidth depends strongly on magnetic inhomogeneities, which are inherent in thin magnetic films and are determined by the quality of the substrates, the specific features of the technique used to fabricate the films, and also by the nonuniformity of the external dc magnetic field in which the films are deposited. We studied the influence of the nonuniformity of the external magnetic field on the magnetic characteristics of Co–P films using three specially prepared samples that have the same thickness of the main layer ($d = 70$ nm) and the same thickness of the nonmagnetic buffer layer ($\delta = 30$ nm). Two of these samples were deposited in a gradient magnetic field, which was produced by two parallel (head-to-head) rectangular cobalt–samarium magnet plates ($40 \times 40 \times 10$ mm in size) placed 30 mm apart (Fig. 7). The samples were located about halfway between the magnets, with the substrate being perpendicular (sample *a*) or parallel (sample *b*) to the magnet surfaces. The third (reference) sample (sample *c*) was deposited in the usual fashion, namely, in a uniform magnetic field applied parallel to the substrate plane.

As one might expect, all magnetic characteristics of films deposited in a gradient magnetic field are significantly more inhomogeneous. The most substantial changes across the film area occur in the uniaxial magnetic anisotropy field H_a, the angle of the easy axis θ_a (Fig. 7), and the magnitude of the unidirectional anisotropy. The average values and standard deviations of some magnetic characteristics of all of the three samples are listed in the table.

From the table, it can be seen that the saturation magnetization, unidirectional anisotropy, and FMR linewidth of sample *a* are about twofold more inhomogeneous than those of sample *b* and are far more inhomogeneous than those of the reference sample. The average FMR linewidth for the reference sample is almost 15 and 30% smaller than that for samples *a* and *b*, respectively.

4. CONCLUSIONS

Thus, our studies have shown that the presence of a nonmagnetic x-ray amorphous buffer layer between the palladium sublayer and the magnetic film significantly increases the mw magnetic permeability of the film, decreases the FMR linewidth, and increases the effective saturation magnetization. The effect is most pronounced if the thickness of the nonmagnetic buffer layer exceeds 20–30 nm.

Our studies have also shown that, for nanocrystalline cobalt films deposited using the technique developed in this work, the FMR linewidth is minimum and, hence, the magnetic permeability is maximum over the decimeter wavelength range if the film thickness is ~20–60 nm. As the film thickness increases, the magnetic permeability decreases due to the skin effect. Therefore, for samples intended for use at lower frequencies, the optimum film thickness increases in proportion to $f^{-0.5}$.

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