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STRUCTURAL AND MAGNETIC PROPERTIES OF NICKEL FILMS DEPOSITED FROM OSCILLATING-ELECTRON DISCHARGE UNDER LOW-ENERGY KRYPTON ION IRRADIATION

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The structural and magnetic parameters of Ni films obtained from a discharge with oscillating electrons are shown to depend on the energy of Kr^+ ions bombarding the film in the course of deposition. The shape of the energy dependence is explained by several factors, including the presence of discrete ionization regions in the discharge, the elastic and inelastic interactions of Kr^+ ions with the film surface, the ratio of fluxes of Ni atoms and Kr^+ ions in the two discharge regimes studied, and the difference in the character of ion interaction with the fcc and hcp phases of nickel.

Studies of the properties of nickel, cobalt, and cobalt-nickel magnetic films, are of interest because of their possible application as magnetic media [1]. The existing methods of film deposition by thermal evaporation offer high deposition rates, but cannot provide sufficiently high purity of the deposit, which hinders the production of homogeneous films with reproducible characteristics. The method of film deposition in a discharge with oscillating electrons [2] can be used under high-vacuum conditions, with the films being continuously cleaned by bombardment with electrons from the discharge region. It is also possible to simultaneously irradiate the films with an ion beam controlled by a bias voltage U_b, thereby affecting both the structure and the magnetic characteristics of the deposited films.

EXPERIMENTAL

We have studied the structure and the magnetic properties of nickel films with thicknesses from 3000 to 6000 Å deposited from a discharge with oscillating electrons onto polished glass substrates. The substrates made of a K-8 grade glass were situated behind the slits of the anode and biased with a negative voltage U_b of 10 to 250 V relative to the anode. As a result, the growing film was bombarded with Kr^+ ions generated near the anode surface due to the gas ionization.

The film thickness was measured by a microinterferometer. The film structure was studied on an ADP-1 diffractometer using monochromatized CuK_α radiation and a detector of the scintillation type. The content of Kr in the film was determined by the IPDO-1 mass spectrometer of the omegatron type. The magnetic properties and the structure of the thin near-surface layer of the film were studied magnetooptically, and the bulk magnetic properties were determined magnetometrically.

Nickel films were obtained at an anodic voltage about 2.5 kV and a magnetic field strength of 275 Oe. The deposition was performed under two discharge regimes, characterized by different pressures of the working intert gas (Kr), potential distributions in the cell, discharge currents J_d, and ion fluxes N_i supplied from the discharge region to the substrate [3]:

Regime I (negative space-charge regime):
$p_{Kr} = (5\text{–}6) \times 10^{-5}$ Torr, $J_d = 0.8\text{–}1$ mA, $N_i = 10^{10}\text{–}10^{12}$ cm^{-2}s^{-1};
Regime II (plasma regime):
$p_{Kr} = (5\text{–}8) \times 10^{-4}$ Torr, $J_d = 3\text{–}4$ mA, $N_i = 10^{13}\text{–}10^{14}$ cm^{-2}s^{-1}.
The flux of metal atoms supplied from cathodes to anodes in both regimes was about 10^{15} cm^{-2}s^{-1}.

RESULTS AND DISCUSSION

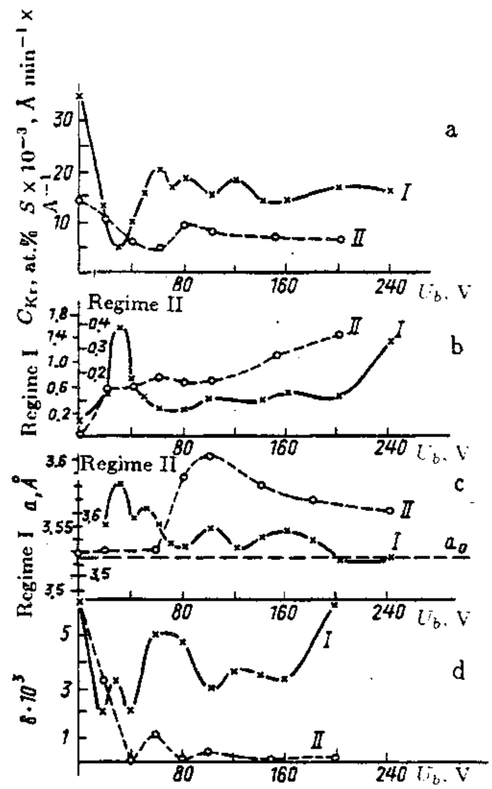

Fig. 1

Figure 1 shows the film deposition rate (S), krypton concentration in the film (C_{Kr}), lattice parameter (a), and the equatorial Kerr effect (δ) versus the bias voltage U_b for nickel films obtained in regime I (curves I). It is seen that the curves exhibit oscillating character. The oscillations of the functions $a = f(U_b)$ and $C_{\mathrm{Kr}} = f(U_b)$ are well correlated with one another, while the oscillations of $S = f(U_b)$ and $\delta = f(U_b)$ are in antiphase with them. The average error of measurements in all plots is 3–5%. The films obtained in this regime had an fcc structure with coherent scattering domains of about 80 Å. The oscillating behavior of the film parameters is caused by discrete ionization regions in the discharge, and by elastic and inelastic interactions of ions emitted from these regions, with the film surface. Figure 2 presents the current passing through the film as a function of U_b for regime I. The curve exhibits well distinguished steps indicating the presence of discrete ionization regions in the discharge. These steps are well pronounced at $U_b < 200$ V. These data agree well with the earlier results [4].

The ions supplied to the film may cause various effects. On the one hand, they produce point defects which serve as condensation and nucleation centers, thus favoring the growth of the film. In this case, a considerable role belongs to inelastic collisions of incident ions with atoms in the growing film and the substrate. On the other hand, the ions may cause reverse sputtering of the film and, hence, a decrease in the overall deposition rate S. Here, elastic interactions become a factor. At low ion energies (from a few to tens of electronvolts), a direct knock-on mechanism is operative. For ions coming from the first ionization region and having energies below a certain threshold value, the ion energy is mostly spent on defect production; only a small proportion of displaced atoms can leave the film. At U_b values close to the threshold energy for Ni sputtering, the S value exhibits a minimum, while C_{Kr} and a reach maximum values. The correlation between C_{Kr} and the lattice parameters of nickel is presumably related to the replacement of Ni atoms in the crystal lattice sites by Kr atoms with the formation of a dilute solid solution of krypton in nickel. Because the atomic radius of Kr is much smaller than that of Ni, the parameter a increases as compared to its value in pure nickel in accordance with the growing krypton concentration in the film.

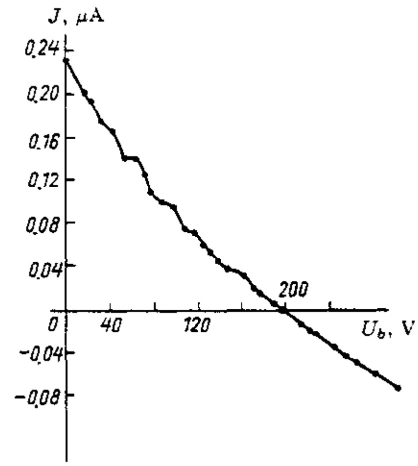

Fig. 2

As the U_b voltage increases, ions from other ionization regions arrive at the substrate. The next minimum of S (with the corresponding maxima of C_{Kr} and a) is observed for ions from second ionization region with energies close to the threshold, and so on. At U_b values corresponding to S_{max}, the elastic interactions are accompanied with inelastic ones. Moreover, ions moving toward the substrate can be partially neutralized by the opposite flow of slow electrons and by a resonance recharging upon interaction of the ground level of Kr ions with the 3d level of Ni. The cross section of resonance neutralization is an oscillating function of the ion energy [5]. This process can lead to the production of structural defects and elastic stress fields stimulating the film nucleation and growth [6]. Part of neutral Kr atoms can be reflected and will not participate in the subsequent sputtering. All this leads to increasing S and decreasing C_{Kr} and a. The fcc type of the film structure remains the same for all films obtained in regime I. This is probably due to the relatively large flux of deposited Ni atoms (about 10^{15} cm^{-2} s^{-1}) compared to the flux of Kr^+ ions bombarding the film (10^{10} to 10^{12} cm^{-2} s^{-1}) during its formation, which leads to a defect concentration that is insufficient to change the stable fcc structure.

The magnetic properties of a thin (about 300 Å) near-surface layer were studied by magnetooptical and optical measurements. For the films obtained in regime I, the frequency dependences of the equatorial Kerr effect (δ) and of the real (ε_1) and imaginary (ε_2) parts of the dielectric constant are similar in shape to analogous dependences observed for polycrystalline fcc nickel. However, the equatorial Kerr effect (which is proportional to the magnetization of the film near-surface region) depends significantly on the deposition regime and the bias voltage U_b. In regime I, the oscillating behavior of $\delta = f(U_b)$ is connected with the shapes of the $a = f(U_b)$ and $S = f(U_b)$ curves depicted in Fig. 1. It is suggested that bombardment of nickel with Kr^+ ions leads to the appearance of nonmagnetic inclusions. The volume fraction of these inclusions depends on U_b and is determined by the Kr concentration in the film.

Figure 1 also shows the curves $S = f(U_b)$, $a = f(U_b)$, and $\delta = f(U_b)$ for Ni films obtained in regime II (curves II). An increase in the pressure renders the ionization regions less pronounced and decreases oscillations in $S = f(U_b)$. No clear correlation is observed between $a = f(U_b)$ and $C_{Kr} = f(U_b)$. The structure of the films obtained in regime II exhibits several characteristic features. In addition to the fcc phase, the films also show a proportion of an hcp phase. At low ion energies (U_b = 20 V), the films are composed almost completely of the fcc Ni phase, with the lattice parameter slightly exceeding that for the bulk Ni (a_0 in Fig. 1 c). However, the X-ray diffraction patterns obtained by the constant-time method and reduced to the amorphous quartz structure (with the substrate scattering intensity subtracted) reveal trace reflections due to an hcp phase. As the ion energy increases to a nearly-threshold level (U_b = 40–60 V), the amount of the fcc phase decreases and that of the hcp phase sharply increases. The measured parameters of the hcp phase are a = 2.60 Å, b = 4.29 Å, c/a = 1.65. Comparison of these values with the data for a massive Ni sample (a = 2.66 Å, c = 4.33 Å, c/a = 1.63) suggests a distortion of the hcp structure. Both phases are also found when the Kr^+ ion energy is increased. Then the fcc lattice parameters depend significantly on the bias voltage, while the parameters of the hcp phase are virtually independent of it. This fact suggests that Kr^+ ions interact predominantly with Ni atoms in the fcc phase. The lack of any clear correlation between the fcc lattice parameters and $C_{Kr}(U_b)$ in Fig. 1 is apparently explained by the presence of two phases (fcc and hcp) in the film, which interact differently with the incident flux of Kr^+ ions. A combined measurement of film grown at $U_b = 200$ V in the symmetric geometry and at a fixed angle of incidence ($\varphi = 10°$) showed that the hcp phase is mostly localized in the near-surface layer of the film, while the fcc phase occurs in deeper layers.

The magnetooptical investigation of the surface of nickel films deposited in regime II showed a zero equatorial Kerr effect for some samples, which indicates a complete absence of magnetization of the light-reflecting nickel surface. A study of surface optical properties revealed that samples with zero magnetization of the near-surface layer have definite features of the frequency dependence of the real (ε_1) and imaginary (ε_2) parts of the dielectric constant in the region of energies $\hbar\omega \sim 1.3$–1.8 eV which indicate that this structure differs from the fcc nickel phase.

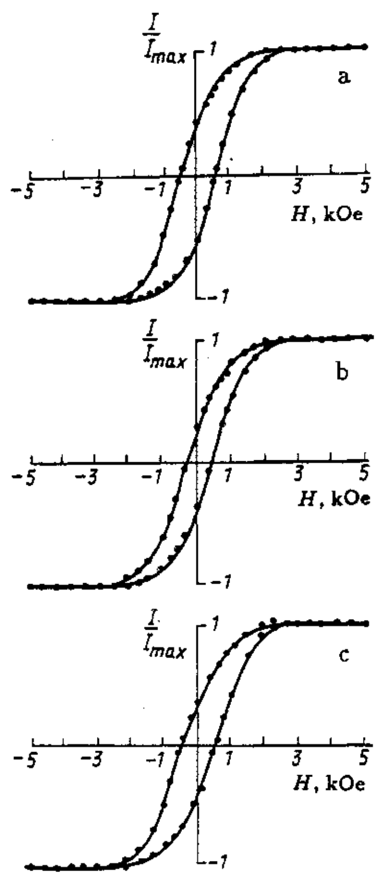

Fig. 3

The bulk magnetic properties of nickel films obtained in regimes I and II were studied on a vibrating-sample magnetometer. All samples exhibited similar hysteresis loops and close values of the bulk magnetization. Comparison of these data with the results obtained by magnetooptical methods (when only a thin near-surface layer about 300 Å thick is probed) shows that the above mentioned hcp nickel phase is localized at the surface and has a zero magnetization. Figure 3 presents the hysteresis loops for two nickel films with nonzero surface magnetizations obtained in regimes I and II (Figs. 3a and b, respectively), and for a film deposited in regime II and having a zero surface magnetization (Fig. 3c).

The appearance of the hcp phase in films deposited in regime II can probably be explained by a greater (as compared to regime I) flux of Kr^+ ions, 10^{13}–10^{14} $cm^{-2}\,s^{-1}$, which becomes comparable with the flux of Ni atoms, 10^{15} $cm^{-2}\,s^{-1}$. There are two possible mechanisms of the hcp structure formation:

(1) The formation of an hcp Ni–Kr solid solution via substitution of Kr atoms for Ni in the lattice sites.

(2) The fcc $\rightarrow$ hcp phase transition stimulated by the internal stress fields around Kr atoms (substituted for Ni in the lattice sites), which can be regarded as defects.

CONCLUSION

The irradiation of a growing nickel film with low-energy Kr^+ ions leads to the formation of a near-surface layer (hcp phase) which differs from the bulk layers (fcc phase) in respect of both structural and magnetic properties. The density of the surface layer depends on the ion energy and the ratio of fluxes of metal atoms and Kr^+ ions supplied to the film.

The films retained their parameters after being stored in air for a year.

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