MAGNETISM AND FERROELECTRICITY

Effect of Nickel on the Magnetic State of Dysprosium in Dy_{1-x}Ni_x-Ni Bilayer Films

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Abstract—This paper reports on the results of investigations into the temperature and spectral dependences of the magnetic circular dichroism in $Dy_{1-x}Ni_x$ –Ni bilayer films prepared through thermal sputter deposition of components under ultrahigh vacuum. The distribution of the components over the layer thickness is examined by Auger spectroscopy. The nickel content *x* in $Dy_{1-x}Ni_x$ layers varies from 0.005 to 0.06. It is shown that, in the temperature range 80–300 K, the contribution made to the magnetic circular dichroism by a $Dy_{1-x}Ni_x$ layer in a bilayer film with a nickel content higher than the threshold value is approximately equal to the magnetic circular dichroism observed in an isolated $Dy_{1-x}Ni_x$ film at temperatures below the temperature of the phase transition to a ferromagnetic state (~100 K). This phenomenon is explained by magnetic ordering in the $Dy_{1-x}Ni_x$ layer of the bilayer film due to the combined effect of two factors, namely, the incorporation of nickel into a dysprosium layer and the presence of a continuous nickel sublayer in the film. © 2003 MAIK "Nauka/Interperiodica".

1. INTRODUCTION

The mutual influence of 3d transition metals and 4frare-earth elements in layered structures has been attracting considerable research attention due to the extensive use of these structures in data recording and storage devices. In recent years, particular interest has been expressed in complex magnetic structures of rareearth metals and their high structural sensitivity to both external and internal actions. One of the factors affecting the magnetic state of rare-earth metal layers in layered structures and superlattices is the interaction of a rare-earth metal either with adjacent layers consisting of 3d transition metals or with 3d metal impurities in a layer of the rare-earth metal. Dysprosium is a convenient object for use in the study of these effects owing to its ability to undergo two magnetic phase transitions separated by a wide temperature interval: (i) the transition from the paramagnetic phase to the spiral antiferromagnetic phase at the Néel temperature $T_{\rm N} = 175$ K and (ii) the transition from the spiral antiferromagnetic phase to the ferromagnetic phase at the Curie temperature $T_c = 85$ K [1–3]. It should be noted that, in this case, the temperature and character of the phase transitions occurring in dysprosium substantially depend on the applied magnetic field [1-4]. The magnetic structure of dysprosium is also strongly affected by the size and structure of the dysprosium sample. In particular, Mulyukov et al. [5] analyzed the influence of the crystallite size on the phase transitions in dysprosium finegrained samples. Shevchenko *et al.* [6] showed that no spiral magnetic structure is formed in dysprosium nanoparticles distributed in an aluminum film.

There are many works concerned with the investigation of dysprosium layered structures containing 3d metals (see, for example, [7–9]). In our previous studies [10, 11], we revealed that 3d impurities at a low content (~5%) in a dysprosium layer substantially affect the temperature and spectral dependences of magneto-optical effects (linear with respect to magnetization), namely, the magnetic circular dichroism and the meridional magneto-optical Kerr effect, in $Dy_{1-x}(NiFe)_{x-1}$ NiFe and $Dy_{1-x}Fe_x$ -Fe bilayer films. (Hereafter, x stands for the weight content.) It was demonstrated that, in the temperature range 80-300 K, the contribution made to the magneto-optical effect by a dysprosium layer in bilayer films containing no more than several weight percent of nickel and iron impurities is constant, temperature independent, and approximately equal to the magneto-optical effect observed in a dysprosium film only at temperatures below the temperature T_c of the phase transition to a ferromagnetic state. Moreover, we established that the magneto-optical effect does not depend on nickel and iron impurities contained in these amounts in dysprosium layers in the case when the adjacent layer of the 3d metal is absent. Consequently, the unusual behavior of the dysprosium magnetic system in the studied samples was explained by two factors: (1) the incorporation of transition metal



Fig. 1. Schematic drawing of a step-shaped film sample: (1) Dy_{1-x} -Ni_x layer, (2) Ni layer, (3) Ni- Dy_{1-x} Ni_x bilayer film, and (4) glass substrate. The sample is separated into three parts along the visual boundaries between them.

atoms at low concentrations into the dysprosium layer throughout its depth and (2) the influence of the spin system of the adjacent transition-metal layer on the magnetic state of dysprosium through the incorporated 3d metal atoms. In order to gain better insight into the nature of the observed effect, it is expedient to examine the magnetic behavior of dysprosium in a bilayer film with an increase in the temperature of the 3d metal layer. However, since dysprosium exhibits a high reactivity, the heating of dysprosium samples leads to contradictory results. One way out is to decrease the Curie temperature of thin nickel films [12]. For a nickel bulk single crystal, the Curie temperature T_c is equal to 631 K [13]. The second important problem is to elucidate how the concentration of 3d metal atoms distributed in the layer of dysprosium affects its magnetic state. Moreover, the changeover from the NiFe alloy films studied in [9, 10] to one-component (nickel) layers should facilitate interpretation of the results. In the present work, the magnetic circular dichroism in $Dy_{1-x}Ni_x$ and Ni single-layer films and $Dy_{1-x}Ni_x-Ni$ bilayer films was investigated as a function of the nickel content x and the thickness of the nickel layer.

2. SAMPLE PREPARATION AND EXPERIMENTAL TECHNIQUE

Samples were prepared by sputtering components from isolated sources under ultrahigh vacuum on an Angara molecular-beam epitaxy setup specially adapted for sputter deposition [14]. Films were deposited on glass substrates (thickness, 0.8 mm; optical quality; surface finish class, 13) at a temperature of 250° C. The sputtered materials were Ni (vacuum melting) and Dy (class DiM 1). The deposition rates for nickel and dysprosium were approximately equal to 0.05 and 2.00 Å s⁻¹, respectively. First, a nickel layer was deposited on the substrate. Then, nickel and dysprosium were sputtered simultaneously. A step-shaped film sample prepared with the use of flaps is represented schematically in Fig. 1. Three samples were obtained by this method under identical conditions: a Ni single-layer film, a $Dy_{1-x}Ni_x$ single-layer film, and a Ni– $Dy_{1-x}Ni_x$ bilayer film. The thickness of each layer in the bilayer film was equal to the thickness of the corresponding single-layer film. The nickel content in the dysprosium layer and the thickness of nickel layers were specified by the conditions of nickel sputtering. We prepared several series of film samples with 50- to 120-Å-thick Ni layers. The thickness of dysprosium layers was varied from 400 to 900 Å. For all samples, the content of each component was determined by x-ray fluorescence analysis.

The magnetic circular dichroism in the spectral range 350-650 nm was measured as the difference between the optical densities $\Delta D = (D_+ - D_-)$ obtained for the right-hand and left-hand circular polarizations of the light wave with respect to the direction of an external magnetic field. The magnetic field was directed perpendicularly to the sample plane and reached 5.0 kOe. The measurements of the magnetic circular dichroism were performed with the use of modulation of a light wave (from the right-hand to lefthand circular polarization) which was described for the first time by Jasperson and Schnatterly [15]. The accuracy in measuring the magnetic circular dichroism was equal to $\pm 10^{-4}$. The temperature investigations were carried out with samples placed in a nitrogen-flow cryostat in the range 80-300 K. The temperature was controlled accurate to within ± 1 K. It is known that, by definition, the magnetic circular dichroism can only be observed in absorbing media. For this reason, neither a glass substrate nor quartz windows of the cryostat contribute to the measured value of magnetic circular dichroism. The magnetic circular dichroism was measured immediately after the samples were prepared.

The distribution of the components over the area and thickness of the samples was determined by Auger electron spectroscopy [16] (sensitivity, 0.3 at. %) after the magneto-optical measurements. The Auger electron spectra of the sample surfaces were recorded on a Riber OPC-2 Auger cylindrical-mirror analyzer with a relative resolution of 0.2%. The secondary electron spectrum was excited by an electron beam with an energy of 3 keV. The electron beam diameter was equal to 5 μ m, and the electron beam current amounted to 100 nA. The high-voltage modulation of the analyzer used was 6.88 eV. For a layer-by-layer analysis, ion sputtering was accomplished using an argon ion beam with an energy of 3 keV at an ion beam current of 600 nA and a rate of approximately 10 Å/min. Since the substrate was prepared from a nonconducting material (glass), it was impossible to obtain spectral data for the filmglass interface due to strong charging of the glass surface. During the layer-by-layer analysis, the Auger signals of elements (Dy, 155 eV; Ni, 848 eV; O, 512 eV; and C, 272 eV) were measured as functions of the time of sputter deposition of the sample with an argon ion beam.

In order to control the degree of oxidation of the dysprosium layers, the electrical conductivity of some samples was measured after the magneto-optical measurements.

3. RESULTS AND DISCUSSION

Figure 2 displays the Auger signals of the main elements in a Ni–Dy bilayer film (x = 0). As can be seen from this figure, nickel impurities are virtually absent throughout the entire thickness of the dysprosium layer, in contrast with the situation discussed in our earlier work [11]. It is worth noting that the oxygen content in the Ni–Dy bilayer film is relatively high, especially at the interfaces with the substrate and the atmosphere. A similar oxygen distribution is observed in a dysprosium single-layer film. In [11], we considered the problem of dysprosium oxidation in sufficient detail. It was demonstrated that, at least during magneto-optical experiments, dysprosium remains in the metallic state and its oxidation to Dy_2O_3 occurs only upon heating to a temperature of ~400°C. This inference is confirmed by the measurements of the electrical conductivity. For example, after the magneto-optical measurements, the electrical resistivity of the 600-Å-thick dysprosium film was equal to $5 \times 10^{-5} \Omega$ cm. In the case when nickel and dysprosium were sputtered simultaneously, nickel impurities were observed throughout the dysprosium layer at a content x ranging from 0.005 to 0.06 depending on the rate of nickel deposition.

The temperature dependences of the reduced magnetic circular dichroism (MCD_T/MCD_{T = 90 K}) for nickel single-layer films of different thicknesses are depicted in Fig. 3. The magnetic circular dichroism of nickel has negative sign, as is the case with the magnetic circular dichroism of permalloy and iron [11]. The dependence of the magnetic circular dichroism on the wavelength is similar to the spectral dependence of the meridional magneto-optical Kerr effect for a nickel single crystal measured by Buschow et al. [17]: the magnetic circular dichroism is nearly constant in the wavelength range 400–700 nm and decreases rapidly with a decrease in the wavelength to 320 nm. Since the magnetic circular dichroism is a linear function of the magnetization, there is a one-to-one correspondence between the temperature dependences of the magnetization and magnetic circular dichroism of the studied samples. These dependences differ from the curves observed for nickel bulk crystals (see [13, Fig. 18.1]). As can be seen from Fig. 3, the smaller the film thickness, the larger the difference. For a nickel film with a minimum used thickness (~50 Å), the magnetic circular dichroism decreases by a factor of approximately two as the temperature increases from 80 to 300 K. For a nickel bulk crystal, the magnetization in this temperature range changes by ~5%. The above temperature behavior of



Fig. 2. Auger signals of the main elements in an Ni–Dy bilayer film: (1) dysprosium, (2) nickel, (3) oxygen, and (4) carbon. $d_{\text{Ni}} = 10 \text{ nm}, d_{\text{Dy}} = 60 \text{ nm}.$



Fig. 3. Reduced temperature dependences of the magnetic circular dichroism $(MCD_T/MCD_T = 90 \text{ K})$ for nickel films of different thicknesses in the magnetic field H = 4.5 kOe at the wavelength $\lambda = 520$ nm. Film thickness: (1) 10 (2, 3) 12, (4) 8, and (5) 6 nm.

the magnetic circular dichroism is similar to the temperature behavior observed for magnetization of very thin layers of nickel in [12] and, hence, can be used to compare the temperature dependences of the magnetic circular dichroism of $Dy_{1-x}Ni_x$ and Ni layers.

For dysprosium films, the temperature and spectral dependences of the magnetic circular dichroism are in good agreement with the data reported in our recent



Fig. 4. (a) Magnetic circular dichroism spectra for a dysprosium film measured in a magnetic field of 5 kOe at temperatures of (1) 93 and (2) 300 K. (b) Spectrum of the off-diagonal component of the optical conductivity tensor for dysprosium [19]. (c) Difference in the optical conductivities for "+" and "–" electrons [20].

paper [11]. Figure 4a shows the magnetic circular dichroism spectra for a dysprosium film measured in a magnetic field of 5 kOe at temperatures of 93 and 300 K. These spectra exhibit the following specific features. First, the magnetic circular dichroism at room temperature has a finite value and is approximately one order of magnitude less than that at a temperature of 93 K. The same ratio was observed for the magnetizations of fine-grained dysprosium samples measured at room and liquid-nitrogen temperatures by Mulyukov et al. [5]. For both temperatures, the magnetic circular dichroism reverses sign at an energy of about 3 eV. It should be noted that the point of sign reversal is somewhat displaced to higher energies with a decrease in the temperature. In the energy range 2–3 eV, there arises a pronounced maximum. In the same spectral range,



Fig. 5. Temperature dependences of the magnetic circular dichroism for (*I*) the Ni film (d = 6 nm), (2) the Dy_{0.995}Ni_{0.005} film (d = 75 nm), and (3) the Dy_{0.995}Ni_{0.005} layer in the Ni–Dy_{0.995}Ni_{0.005} bilayer film according to (a) experimental data and (b) reduced magnitudes MCD_T/MCD_{T = 90 K}. H = 4.5 kOe, $\lambda = 520$ nm.

Knyazev and Noskov [18] observed a maximum in the optical conductivity $\sigma = nkv$ for dysprosium. The magnetic circular dichroism spectra for different samples of dysprosium are similar to one another. Small differences observed in the vicinity of the maximum can be associated with imperfection of the films. However, the point at which the magnetic circular dichroism reverses sign remains the same for all the studied samples. The curve of the magnetic circular dichroism is closely similar to that of both the off-diagonal component of the optical conductivity tensor σ_{xy} given in [19] (Fig. 4b) and the difference in the optical conductivities determined in [20] for "+" and "-" electrons from the calculated densities of states of these electrons (Fig. 4c). As can be seen, the point of sign reversal for the above effects is displaced toward an increase in the light wave

energy upon changing over from the magnetic circular dichroism (Fig. 4a) to the off-diagonal component of the optical conductivity tensor (Fig. 4b) and then to the difference in the optical conductivities (Fig. 4c). Note that the curves depicted in Figs. 4a and 4b differ only slightly.

The magnetic circular dichroism of the Dy-Ni bilayer film, for which the Auger spectrum is displayed in Fig. 2, is equal to the sum of the magnetic circular dichroisms of its constituent layers of dysprosium and nickel at all the temperatures and wavelengths used in the measurements. A similar situation is observed at a low nickel content in a dysprosium layer. Figure 5 shows the temperature dependences of the magnetic circular dichroism for an Ni film (curve 1), a $Dy_{0.995}Ni_{0.005}$ film (curve 2), and a $Dy_{0.995}Ni_{0.005}$ layer in the Ni–Dy_{0.995}Ni_{0.005} bilayer film (curve 3). Curve 3 was obtained as the difference between the values of the magnetic circular dichroism measured in the bilayer film and the nickel single-layer film prepared in the same cycle of deposition. It can be seen from Fig. 5 that curves 2 and 3 almost coincide with each other. This suggests that nickel impurities at a content of 0.5% have no effect on the magnetic properties of dysprosium either in the form of a single-layer film or in the case when this film is applied on the nickel layer. However, at a nickel content $x \approx 0.01$ (i.e., 1.0%), the magnetic circular dichroism of the bilayer film differs from the sum of the magnetic circular dichroisms of the $Dy_{0.99}Ni_{0.01}$ and Ni layers (Fig. 6). For the $Dy_{0.99}Ni_{0.01}$ layer in the bilayer film at room temperature, the magnetic circular dichroism has a relatively large value. The sign of the observed effect corresponds to the sign of the magnetic circular dichroism in a single-layer film of the same composition. In the temperature range 120-300 K, the magnetic circular dichroism in both cases does not depend on the temperature. As the temperature decreases below 120 K, the magnetic circular dichroism in the $Dy_{0.99}Ni_{0.01}$ layer of the bilayer film increases in the same manner as in the Dy_{0.99}Ni_{0.01} single-layer film. It is clearly seen that the temperature dependences of the magnetic circular dichroism in nickel (curve 1) and $Dy_{0.99}Ni_{0.01}$ (curves 2, 3) films differ significantly. With a further increase in the nickel content x, the similarity of the temperature dependences of the magnetic circular dichroism for $Dy_{1-x}Ni_x$ single-layer films to those for single-layer films of the same composition but applied on the nickel layer gradually becomes less pronounced. In the entire temperature range covered, the magnetic circular dichroism of a $Dy_{1-x}Ni_x$ layer (x = 0.02) applied on the nickel layer is close to that measured for an isolated $Dy_{1-x}Ni_x$ layer at a temperature of 90 K. It is worth noting that the temperature dependence of the magnetic circular dichroism of the former $Dy_{1-x}Ni_x$ layer is very similar to that of the nickel film (Fig. 6, curve 4). An increase in the nickel content x also leads to some variations in the spectral dependences of the magnetic circular dichroism of the $Dy_{1-x}Ni_x$ layer



Fig. 6. Reduced temperature dependences of the magnitude of the magnetic circular dichroism $(MCD_T/MCD_{T=90 \text{ K}})$ for (1) the Ni film (d = 10 nm), (2) the $Dy_{0.95}Ni_{0.05}$ film (d = 60 nm), (3) the $Dy_{0.98}Ni_{0.02}$ layer in an Ni– $Dy_{1-x}Ni_x$ bilayer film, and (4) the $Dy_{0.95}Ni_{0.05}$ layer in an Ni– $Dy_{1-x}Ni_x$ bilayer film. H = 4.5 kOe, $\lambda = 520$ nm.

applied on the nickel layer. For example, the point at which the magnetic circular dichroism reverses sign is displaced to higher energies.

Summing up the above results of the magnetic circular dichroism measurements, we can draw the following conclusions: (i) nickel impurities in a dysprosium layer substantially affect the temperature and spectral dependences of the magnetic circular dichroism only in the case when the $Dy_{1-x}Ni_x$ layer is in contact with a nickel continuous layer; (ii) the threshold content of nickel in the dysprosium layer at which the temperature dependence of the magnetic circular dichroism of the $Dy_{1-x}Ni_x$ single-layer film ceases to be similar to that of a single-layer film of the same composition but applied on the nickel layer amounts to ~2%; and (iii) at a nickel content of approximately 2%, the temperature dependence of the magnetic circular dichroism of the $Dy_{1-x}Ni_x$ layer in a bilayer film is very similar to that of the nickel film. The last result is in agreement with the data obtained in our previous works [10, 11] concerned with the study of $Dy_{1-x}(Ni_{80}Fe_{20})_x$ -NiFe and $Dy_{1-x}Fe_x$ -Fe bilayer films with a content of $Ni_{80}Fe_{20}$ or Fe in the dysprosium layer approximately equal to 3%. As was shown in [10, 11], in the temperature range 80-300 K, the magnetic circular dichroism in Ni₈₀Fe₂₀ and Fe layers does not depend on temperature and the contribution made to the magnetic circular dichroism by a dysprosium layer containing nickel and iron impurities and involved in a bilayer film is also temperature independent. The magnetic circular dichroism observed at temperatures considerably above the temperature of ferromagnetic ordering of a dysprosium bulk crystal ($T_c = 85$ K) can be associated with magnetic ordering in the $Dy_{1-x}Ni_x$ layer. The sign of the magnetic circular dichroism indicates that the magnetic moment of the $Dy_{1-x}Ni_x$ layer in the situation under investigation is aligned parallel to the magnetic moment of the nickel layer.

The magnetic polarization of 4f electrons of rareearth metals on the surface of 3d metals was analyzed earlier theoretically and observed experimentally in very thin layers. In particular, Carbone et al. [21] observed the polarization of 4f electron spins in dysprosium monoatomic layers applied on the surface of a single crystal or a 3d metal film. The spin moments of dysprosium were antiparallel to those of the crystal surface or the 3d metal layer. Judging from the strong dependence of the temperature behavior of the magnetic circular dichroism in the $Dy_{1-x}Ni_x$ layer on the nickel content x, it can be assumed that, in the case under consideration, impurity nickel atoms and their bonds with a nickel continuous magnetically ordered layer are responsible for the effect of the 3d metal on the magnetic state of dysprosium at a large depth of the dysprosium layer.

Matveeva and Egorov [20] noted that, in the spectrum of dysprosium (Fig. 4c), excitations at low energies dominate in the system of "-" electrons, whereas excitations at high energies dominate in the system of "+" electrons. A drastic increase in the conductivity at the expense of "+" electrons near 4 eV corresponds to the threshold of 4f excitations. The magnetic circular dichroism and the off-diagonal component of the optical conductivity tensor σ_{xy} should also reverse sign at about the same energy, which is observed in the experiment. Therefore, the displacement of the point at which the magnetic circular dichroism passes through zero for the $Dy_{1-x}Ni_x$ single-layer film applied on the nickel layer is most likely associated with the transformation in the electronic structure of either the $Dy_{1-x}Ni_x$ layer or the film as a whole.

As is known [3], the magnetic structure of heavy rare-earth metals, including dysprosium, is determined primarily by the exchange interaction of conduction electrons with 4f electrons. The introduction of nickel atoms into a dysprosium layer leads to changes in the system of conduction electrons. The magnetic moments of nickel atoms can be polarized because of the presence of a continuous, magnetically ordered nickel layer. The threshold weight content of nickel at which dysprosium at room temperature undergoes magnetic ordering is approximately equal to 2%. With due regard for the atomic weights of the components, this value corresponds to ~5 vol %. It can easily be shown that, in the case of a uniform distribution of nickel over the dysprosium layer, the second coordination shell of each dysprosium ion contains a nickel ion with a spin aligned parallel to the magnetic moment of the nickel layer. The hybridization of Ni *d* states with Dy *f* states can stimulate magnetic ordering in the Dy_{1-x}Ni_x layer. On the other hand, it can be assumed that the sample as a whole is characterized by a single system of conduction electrons. Consequently, all conduction electrons are polarized, including those provided by the Ni *s*, Dy *p*, and Dy *d* states. In this situation, spins of Dy 4*f* electrons undergo ordering due to the interaction with polarized band electrons. In both cases, the temperature dependences of the magnetization and, hence, the magnetic circular dichroism in the Dy_{1-x}Ni_x layer should correlate with those of the nickel layer and with the nickel content in the dysprosium layer.

Moreover, nickel impurities bring about insignificant distortions in the dysprosium lattice because of the differences between the atomic and ionic radii ($r_{\rm Dy} =$ 1.773 Å, $r_{\rm Dy^{3+}} = 1.07$ Å, $r_{\rm Ni} = 1.25$ Å, and $r_{\rm Ni^{2+}} =$ 0.72 Å). These distortions can affect the magnetic structure of the layer due to strong spin–orbit coupling in dysprosium and competition between exchange interactions in different coordination shells. However, in this case, the magnetic ordering in the Dy_{1-x}Ni_x layer should also be observed in the absence of the nickel layer, which is inconsistent with the available experimental data.

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