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### Magnetic properties of Fe-Bi films

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Abstract. In the paper the results of experimental studies of film structures in the Fe-Bi system are presented. By the method of electron magnetic resonance, it is shown that for bilayer structures the magnetic state depends on the order of deposition of the magnetic and nonmagnetic layers. In the three-layer structures, the effect of exchange bias was found, the magnitude of that depends on the thickness of the bismuth layer.

#### 1. Introduction

Magnetic nanoscale layered structures with a semi-metallic interlayer are poorly studied objects, and are of considerable interest to the physics of the condensed state. Film structures with an intermediate nonmagnetic bismuth layer have been studied previously. In the structure of CoFe/Bi/Co [1], it was found that the interlayer interaction exhibits two oscillation periods of 9 and 25 nm. The study of Co/Bi/Co samples [2] also confirmed the presence of a exchange between the magnetic layers in a wide range of interlayer thicknesses (from 0.2 to 50 nm). Here, the dependence of the coercive force  $(H_{\rm C})$  and the saturation magnetization  $(H_{\rm S})$  is oscillating character with different oscillation periods. In the film structures of the Fe – Bi system [3], it was found that if the thickness of the iron layer was less than 1.5 nm, then the Fe/Bi films had perpendicular magnetic anisotropy, while at greater thicknesses of the iron layer the magnetization lay in the film plane. In the FeNi/Bi/FeNi trilayer structures [4, 5] with a ferromagnetic layer thickness of  $t_{FeNi} = 10$  nm, no change of anisotropy signs in the film plane are observed. In this case, it was established that there is an oscillation of interlayer exchange with a period  $t_{Bi} \sim 8$  nm and a change in the sign of anisotropy in the region  $t_{Bi} \sim 15$  nm.

Depending on the production technology, either solid solutions [6] at high speeds and high deposition temperatures (Fe<sub>x</sub>Bi<sub>1-x</sub> films), or film structures at low deposition rates (Fe/Bi) [3] are obtained. In the first case, as a rule, a magnetic state of the "spin glass" type is realized, and in the second case, the spectrum of manifestations is much wider.

#### 2. Experimental

Films were obtained by thermal evaporation at a base vacuum of P  $\sim 10^{-6}$  Tor. Iron was chosen as the magnetic material due to the fact that in our case it is easy to control the formation of metastable iron modifications. Moreover, among the semimetal elements the bismuth is distinguished by the fact that it practically does not form chemical compounds with 3d-metals [7]. During deposition, a magnetic field of ~ 200 Oe was applied along to axis of easy magnetization in the film plane. In one cycle, films of Fe/Fe, Fe/Bi and Bi/Fe compositions were sputtered onto glass substrates. Fe/Bi/Fe films were



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deposited independently in another cycle. For all films, the magnetic layer thickness was  $t_{Fe} \approx 10$  nm, and the bismuth thickness was  $t_{Bi} = 15$  nm for bilayer structures, and  $t_{Bi} = 3.5$ , 4.5, 6, 8, 10, 12 nm for three-layer films.

The thickness of the layers was determined by X-ray spectroscopy. Electron-microscopic measurements showed that the layers are continuous in are and their composition corresponds to nominal. No traces of the presence of 3d-metal – bismuth compounds were found. The presence of iron oxides is also not detected. A coating of either Ag or Cu with a thickness of 10–20 nm

was applied on the films from above. The surface structure of the films was studied on a Veeco Multi Mode atomic force microscope (AFM) (resolution 1 nm) (figure1). It has been established that the surface roughness height does not exceed 2.5 nm, it means that there is no direct contact between the ferromagnetic iron layers. The magnetization was measured on the MPMS-XL SQUID-magnetometer. For magnetostatic measurements, the external field lay in the film plane and was directed along the induced easy axis. To measure the resonance properties, the Bruker E 500 CW EPR EPR spectrometer was used, which operates at a frequency of  $f_{MWF} = 9.48$  GHz.



**Figure 1.** The morphology of the Bi/Fe film, obtained by the AFM method.

#### 3. Results and discussion

By study film structures in the Fe-Bi system, it was found that the magnetic behavior of bilayer films strongly depends on the order of deposition of the magnetic and non-magnetic layers [8]. Most revealing is manifested in magnetic resonance parameters. When the magnetic field lies in the film plane, a single absorption peak is observed, the position of which depends on the sequence of deposition of the layers (see figure 2).



**Figure 2.** Magnetic resonance spectrum of the films: **a**: 1 - Fe, 2 - Fe/Bi, **b**: Bi/Fe. The magnetic field is along the easy direction in the plane of the film. T = 300 K.

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In the case when the external magnetic field is directed perpendicular to the plane of a bilayer Bi/Fe film, two absorption lines are observed, and for the other two compositions (Fe/Bi and Fe) we still have a single absorption line with similar values of resonance fields.

These data indicate that, in the case of the Bi/Fe structure, in the process of sputtering, another subsystem arises with a stronger anisotropy. So for films in the Co-Bi system, it was found that the structure of  $[Co/Bi]_{19}/Co$  [9] with a total thickness of < 100 nm and different thicknesses of the Co and Bi layers does not produce a sharp layered structure, and a sequence of bismuth layers with interspersed with cobalt granules. Based on the fact that the iron melting temperature is equal to  $\approx$ 1812 K, a melting temperature of bismuth is equal to  $\approx$  545 K, we can assume that in the case of Bi/Fe films strongly heated by high energy iron ions incident on the layer of bismuth, which is fusible. Iron penetrates deep enough into the thickness of the bismuth layer. As a result, a layer of nanoscale iron granules is formed. These circumstances affect the resonant properties of bilayer systems. In the case of Fe/Bi, the modified iron subsystem does not form, moreover, as can be seen in figure 2a, the influence of the interface on the Fe – Bi interface practically does not change the properties of the iron layer (the resonant fields of lines 1 and 2 differ very little). For the Bi/Fe film, the presence of a granular layer is clearly manifested. These data are confirmed by magnetostatic measurements on bilayer films [8]. The coercive force depends on the sequence of deposition of the layers (the difference is almost 5 times), and the saturation magnetizations coincide within the experimental error. This is especially evident from the resonance spectrum in geometry, when the external magnetic field is directed perpendicular to the film plane (perpendicular geometry). Here the magnetic resonance spectrum consists of two lines. It is established that one line lies in the region of the fields inherent in the magnetic resonance of the iron film, and the other line lies in the field of much larger fields. Such behavior is explained if one considers that sputtering occurs in a magnetic field, as a result of which highly anisotropic iron granules [10] are mainly oriented along the induced easy axis. In perpendicular geometry, the granular subsystem manifests itself, for which anisotropy is added, which puts down the magnetic moment of the granules in the film plane. It leads to a shift of the resonance in the region of large fields.

It is clear that this feature should manifest itself in the magnetic properties of multilayer structures. Figure 3 shows the magnetization curves of three-layer Fe/Bi/Fe films with different thicknesses of a semi-metallic interlayer ( $\sigma_{Fe} = M_{Fe} \cdot t_{Fe}$  is the magnetic moment per unit surface of the film). At helium temperatures, the magnetization curves have a biased form. Moreover, both the coercive force and the magnitude of the exchange bias depend on the thickness of the bismuth layer. At room temperature, the bias disappears.



Figure 3. Dependence of the exchange bias  $H_E$  on the thickness of the nonmagnetic interlayer in Fe/Bi/Fe films at T = 4.2 K.

The exchange bias field is usually defined as  $H_E = (H_{C2} + H_C1)/2$  (see figure 3, part 1), where  $H_{C1}$  and  $H_{C2}$  are the coercive fields of the magnetization curve. In our case,  $H_E < 0$ , it means that the exchange interaction between the pinning and the reversible layers is antiferromagnetic. As a pinning layer, apparently, there is a granulated iron sublayer formed at the Bi-Fe interface. Figure 4 shows the dependence of the exchange bias field on the thickness of the nonmagnetic bismuth layer. It can be seen that there is a maximum of the curve in the vicinity of  $t_{Bi} = 4.5$  nm; with a further increase in the

thickness of bismuth, a decrease in  $H_E$  occurs. If we propose a model for our situation, then the system can be represented in the composition  $Fe_1/Bi/Fe_{Gr}/Fe_2$ , where  $Fe_{Gr}$  is a granular iron subsystem.

**Figure 4.** Dependence of the exchange bias  $H_E$  on the thickness of the nonmagnetic interlayer in Fe/ Bi/Fe films at T = 4.2 K.

As the experiment shows,  $M_{Fe1} \approx M_{Fe2} + M_{FeGr}$ , provided that the  $M_{Fe2}$  and  $M_{FeGr}$  subsystems interact ferromagnetically. The interaction through a bismuth layer is antiferromagnetic. The features of anisotropy will be determined by the granular subsystem. This situation is to some extent similar to that considered in [11] for a bilayer system of spin glass (SG)/ferromagnet (FM), where the spin glass layer plays the role of a pinning layer. Here a strong influence of the cooling field and temperature on the magnitude of the exchange bias was found. The current state of the theory of exchange bias in nanostructures is presented in [12], where an antiferromagnetic layer is considered as a pinning layer. However, for such exotic situations as in [11] or in our case, a strict theory is not constructed. The dependence of the exchange bias on the thickness of the bismuth layer is qualitatively understandable. At small thicknesses of bismuth during the deposition of iron, it seems to penetrate to a depth of  $t_{max}$  = 4.5 nm. At bismuth thicknesses  $t_{Bi} < t_{max}$ , the thickness of the granular subsystem of iron increases, which reaches a maximum value at t<sub>max</sub>. At the same time, the volume and anisotropy of the granular subsystem grows. With a further increase in  $t_{Bi}$ , the effect of weakening the interlayer interaction begins to predominate, which leads to a decrease in the exchange bias. When magnetized, the pinning layer does not manifest itself as a step, because its fraction is rather small. It has strong anisotropy (the anisotropy of a granule with a diameter of d ~ 5.5 nm is  $K \approx 1.3 \cdot 10^6 \text{ erg/cm}^3$  [10]) and a well-defined saturation in fields H < 1.5 kOe does not occur, and there is a "paraprocess" (figure 2).

#### 4. Conclusion

As a result of the research, it has been established that in bilayer film structures the magnetic state depends on the sequence of deposition of the ferromagnetic layer of iron and the non-magnetic layer of bismuth. At the same time, for the structures of the composition Bi/Fe, a subsystem of granulated iron is formed, possessing strong magnetic anisotropy. The existence of such a granular subsystem leads to the nonequivalence of magnetic layers in multilayer film structures. One of the manifestations of this effect is the appearance of an exchange bias, depending on the thickness of the nonmagnetic semimetal layer.

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