MODIFICATION OF THE STRUCTURE AND MAGNETIC PROPERTIES OF Gd FILMS DOPED WITH Co

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Abstract. The article presents experimental data on the structure and magnetic properties of films of the Gd–Co system with a low Co content (up to 20 at %) obtained by magnetron sputtering. It was found that the films of pure Gd differ in structural heterogeneity, expressed in the presence of crystalline hcp and fcc phases and presumably an X-ray amorphous phase with a large dispersion in the size of nanocrystallites. The ferromagnetism of the crystalline state and the metabolic frustration of the nanocrystalline phase ensure the asperomagnetic state of the films. Amorphization, which increases with an increase in the Co content, leads to the formation of a more homogeneous distribution of Gd atoms in the medium and provides a gradual transition to a ferromagnetic state, but with a reduced Curie temperature.

Keywords: *gadolinium, cobalt, films, structure, magnetic properties, ferromagnetism, asperomagnetism*

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INTRODUCTION

Film structures based on alloys of heavy rare-earth (R = Gd, Tb, Dy, Ho) and 3d-transition metals (T = Fe, Co, Ni) are an object of increased research interest due to the possibility of wide variation of their magnetic structure and macroscopic magnetic properties. This is due to individual features of exchange interactions and magnetic anisotropy in combination with high mutual solubility of components of such bi-nary systems [1]. In this case, R-elements as carriers of localised magnetism within the framework of cos-venous exchange largely set the character of the magnetic structure, while T-elements, being zone magnetics, contribute to the increase in the temperature of magnetic ordering. As a result, R-T films in wide concentration and temperature intervals are often characterised by unique noncollinear magnetism in spero-, aspero- or sperimagnetic variants [2-5]. It can be considered as a certain basis for the functionality of media for spintronics devices and microelectromechanical systems [6-9].

The structural specificity of R-T films consists in a pronounced tendency to amorphisation, which is caused, on the one hand, by a significant difference in the atomic radii of rare-earth elements and iron group elements, and, on the other hand, by the limited mobility of atoms on the substrate [10-11]. As a result, instead of a set of intermetallic compounds that distinguish R-T alloys [12-14], films are characterised by a wide range of solid solutions, which allows us to analyse in detail the regularities of the formation of magnetic properties of these systems. To date, the properties of films in which the concentration of T elements exceeds 50% and the magnetic order exists above room temperature have been sufficiently studied. An important stimulus for this is

the prospect of practical applications of amorphous films as magneto-solid or magnetostrictive media, as well as carriers of perpendicular magnetic anisotropy, on which high-density information recording can be based [15].

In the other part of the concentration range, *R* - *T* -compositions *T* -subsystem does not contribute to spontaneous magnetization. This circumstance reflects the band type of magnetism of 3 *d* -elements, the parameters of which are appropriately modified by the conduction electrons of the *R* -subsystem [16]. Such films have not found direct application in traditional areas of microelectronics and are poorly studied. However, to some extent, they may be of interest for low-temperature spintronics as media with complex and adjustable magnetic structure. Additionally, they can provide further information about the patterns of superposition of collectivized and localized types of magnetism. This work is dedicated to a systematic experimental study of the magnetic properties of $Gd_{100-x}Co_x$ films in the low-cobalt composition region ($x < 50$), when Co effectively acts as an alloying amorphizing additive, in order to compare them with the structural changes introduced by Co.

SAMPLES AND RESEARCH METHODS

Film samples of $Gd_{100-x}Co_x$ (x = 0–20 %) with a compositional step of about 3 at.% were obtained by magnetron sputtering of single-component targets in an argon atmosphere at a pressure of $1 \cdot 10^{-3}$ Torr. The formation of films occurred in the presence of a technological magnetic field with a strength of 250 Oe, oriented in the plane of the substrates, which were Corning cover glasses. Protection of the samples from surface oxidation was provided by an additional Ta layer. The nominal thicknesses of the Gd–Co and Ta layers were 100 and 5 nm, respectively. The composition was varied by changing the ratio of deposition rates of Gd and Co, which in turn were regulated by appropriately selecting the electrical powers on the magnetrons. The elemental composition of the films was controlled using a Nanohunter X-ray fluorescence spectrometer. Structural data were obtained by X-ray diffraction on a PANalytical Empyrean instrument using $Co_{K\alpha}$ radiation. Magnetic measurements were carried out on a PPMS DynaCool setup with a "vibrating sample magnetometer" option with magnetic field variation within ±70 kOe, which was oriented in the film plane, and in the temperature range from 5 to 350 K.

RESULTS

1. Structural analysis

Characteristic diffractograms of $Gd_{100-x}Co_x$ films of several compositions are shown in Fig. 1. Their analysis allows for the following conclusions. For the pure Gd sample, the diffractogram contains a series of lines that are most likely formed by reflections from planes of the (100) type in the HCP crystal lattice, characteristic of this metal [17]. The exception is the diffraction peak marked with a red dashed line ($2\theta \approx 34°$). It may indicate the presence of crystallites with an FCC lattice in the sample, textured according to the (111) type. It should be noted that the possibility of forming "cubic" Gd in films obtained with a specific combination of technological parameters, such as working gas pressure, deposition rate, and substrate temperature, is indicated, in particular, in [18]. However, there is another interpretation for this fact – the presence of Gd_2O_3 oxide in the sample. This compound, by its crystal structure (parameter $a = 0.531 \pm 0.002$ nm [19]), is practically identical to "cubic" Gd ($a = 0.533 \pm 0.001$ nm [20]), which does not allow for unambiguous phase identification solely based on the presented X-ray data. However, jumping ahead, it can be noted that the analysis of magnetic properties of this sample indirectly indicates the absence of any significant amount of oxide phase in it. Another distinctive feature of the considered diffractogram is the presence of a broad maximum in the angular region where the main diffraction lines of crystalline Gd are located. This indicates the presence of a large X-ray amorphous component in the sample and, in our opinion, is formed not only by the glass substrate but also by a metallic X-ray amorphous phase, which is highly likely present in the film. Due to the small volume, it is not possible to directly isolate the contribution from the film, and the corresponding conclusion was made based on the subsequent analysis of magnetic properties. This analysis was performed based on the fact that pure Gd films are characterized by structural heterogeneity and include, in significant quantities, at least two crystalline phases characterized by hexagonal and cubic symmetry, and a phase identified as X-ray amorphous.

Fig. 1. Diffractograms of $Gd_{100-x}Co_x$ films.

The diffraction pattern undergoes certain changes within the binary Gd-Co system. First, with the introduction of a relatively small amount of Co, the intensity of all identified lines decreases, indicating increasing amorphization of the films. Note that this pattern also applies to the line formed by the FCC lattice. This would hardly be the case if it reflected the presence of Gd oxide in the film. Second, there is a redistribution of diffraction intensity on various crystalline planes, indicating a modification of the crystalline texture initiated by Co. Already at the initial stage of doping, the (002) line virtually disappears, but the previously shadowed (101) line becomes clearly defined. This texture variant proves to be the most resistant to amorphization, which is fully realized at $x \geq 20$. The latter fits well into the known scheme of metallic glass formation, which defines the optimal atomic ratio between the base metal and the amorphizing additive as 80:20. Of course, the conclusion about amorphization is made based on the facts of intensity reduction and disappearance of diffraction lines. On the actual "amorphous" halo, changes are practically imperceptible due to the aforementioned shadowing effect of the relatively thick amorphous substrate.

2. Magnetic properties of Gd film

The magnetic properties of the Gd film are characterized in Fig. 2, which shows the temperature dependences of magnetization $M(T)$ and inverse paramagnetic susceptibility $\chi^{-1}(T)$, and also the hysteresis loop $M(H)$, measured at $T = 5$ K. The $M(T)$ dependence was determined in a magnetic field with a strength of 100 Oe during monotonic heating of the sample. The sample was cooled to the initial temperature ($T = 5$ K) in a magnetic field with a strength of 70 kOe, which, like the measuring field, was co-directed with the technological field present during the production of the films.

Fig. 2. Magnetic properties of the Gd film: a) temperature dependencies of magnetization (curve 1) and inverse paramagnetic susceptibility (curve 2); b) hysteresis loop measured at a temperature of 5 K.

From the form and quantitative characteristics of the dependency $M(T)$ it can be concluded that Gd films demonstrate properties significantly different from those of the bulk metal. Thus, the magnetization value at $T = 5$ K is about 500 G, which is four times less than the tabulated value of spontaneous magnetization (1950 G). At the same time, the geometry of the experiment and the magnetic history do not allow attributing this difference to the self-demagnetization effect. Additionally, the $M(T)$ curve has a concave character untypical for ferromagnets, although the Curie temperature ($T_C \sim 280$ K) is only slightly lower than the magnetic ordering temperature of bulk Gd (293 K) [21]. It should be noted that a similar ratio in the values of T_C was registered earlier in studies of relatively thick Gd films [22], but other features in the formation of $M(T)$ from those mentioned above have not been covered in the literature.

Previously [23], the low value of $M(T = 5$ K) was associated by us in part with the asperomagnetism of Gd films, which is effectively realized in the superposition of ferromagnetic and speromagnetic structures, the carriers of which apparently are crystalline [24-26] and a significant part of the X-ray amorphous phase, respectively. The cause of speromagnetism may be the orientational frustration of local magnetic moments, due to the dispersion of interatomic distances, and, in accordance with the RKKY model, variation of indirect exchange bonds in a highly defective structural state. Usually, the noncollinear nature of the magnetic structure in R - T systems with anisotropic rare-earth ions (Tb, Dy) [27, 28] is associated with the disorientation of local easy magnetization axes. In the case of R = Gd with its spherical 4f-electron shell, this circumstance cannot be the determining cause of asperomagnetism.

In favor of the stated position, there is also a relationship $M(H)$ (Fig. 2b) that is atypical for films of low-anisotropic magnets. The high magnetic susceptibility in strong fields and the absence of magnetic saturation in a sufficiently strong magnetic field (up to 70 kOe) indicate not only heterogeneity but also significant "rigidity" of the magnetic structure, which can naturally be associated with interaction of exchange nature. At the same time, the low-field part of the hysteresis loop, including the presence of some hysteresis (coercive force $H_c \sim 600$ Oe), may reflect the dispersion of a relatively small crystalline magnetic anisotropy. According to some data [29], it is still observed in Gd at low temperatures.

Within the framework of structural heterogeneity, an explanation can also be given for the concavity of the $M(T)$ curve. For this, it is necessary to assume that the X-ray amorphous phase itself is heterogeneous and contains a set of structural elements of different lengths and, accordingly, with different degrees of atomic

order. Some of them are speromagnetic, and some are ferromagnetic, i.e., characterized by a convex dependence $M_i(T)$, but with an individual Curie temperature T_{Ci}, depending on the level of structural ordering. Then the resulting magnetization

$$M(T) = \Sigma n_i M_i(T) \qquad (1)$$

with appropriate sets of T_{Ci}, parameters n_i, which determine the concentration of elements, and a large set of elements themselves, will have a smooth and concave appearance. At the same time, the upper limit for T_{Ci} is the Curie temperature of the crystalline phase, which apparently determines the high-temperature part of the $M(T)$ dependence, which essentially has a "ferromagnetic" character.

In addition, Fig. 2a shows the temperature dependence of inverse paramagnetic susceptibility, which, using the standard methodology [23], allows estimation of the magnetic moment m of Gd ions. For this purpose, the volume of the film sample was determined by the measured thickness and area, and the calculated material density was determined from X-ray data. As a result, m = 7.7 μ_B was obtained. As can be seen, this value is close to the theoretical magnetic moment of a free Gd atom (7 μ_B). Thus, this result can be considered as confirmation that the films contain virtually no gadolinium bound in oxide, and the (111) line in the diffractogram belongs to "cubic" Gd, which is paramagnetic in the studied temperature range, taking into account the film production technique [30, 31].

3. Magnetic properties of $Gd_{100-x}Co_x$ films

The modification of magnetic properties of Gd films occurring when Co is introduced up to 20 at.% is illustrated in Fig. 3. As can be seen, doping leads to a cardinal change in the character of the temperature dependence of magnetization (Fig. 3a). As the Co content increases, the $M(T)$ dependence gradually transitions from concave to convex. This is accompanied by a multiple increase in the value of $M(T = 5\ K)$ and a decrease in the Curie temperature.

Fig. 3. Temperature dependencies of magnetization (a), measured in a magnetic field of 100 Oe, and magnetization curves (b), measured at a temperature of 5 K, for Gd–Co films of different compositions.

Within the framework of the above interpretation of the properties of pure Gd, the observed changes can be considered as a consequence of increased homogeneity of the magnetic structure. The X-ray data presented above indicated that doping leads to amorphization of crystalline Gd, which is practically completed at x = 20. The decrease in the crystalline phase fraction is most likely responsible for the modification of the high-temperature ($T > 150$ K) section of the $M(T)$ dependence, which actually disappears when amorphization is complete. Along with this, certain structural changes apparently occur within the X-ray amorphous phase as well. In our opinion, they are responsible for the modification of the $M(T)$ dependence in the low-temperature region. It can be assumed that in the final amorphous structure, the dispersion in interatomic distances of the gadolinium subsystem is significantly less than in the intermediate nanocrystalline variant. The formation of such a more homogeneous state in some sense reduces the exchange frustration of the system and leads to a certain value of the Curie temperature (~130 K) in most of the film volume. As a result, the $M(T)$ dependence at x = 20 acquires the characteristic appearance of ferromagnets.

The magnetization curves shown in Fig. 3b also reflect the structural changes occurring during doping. First, with an increase in Co content and amorphization progression, the maximum magnetization value achieved in the field H =70 kOe increases. Second, the high-field magnetic susceptibility decreases. Both indicate a "facilitation" of the magnetization process associated with a decrease in the proportion of the exchange frustrated phase.

Fig. 4. Temperature dependences of high-field susceptibility of $Gd_{100-x}Co_x$ films of different compositions.

Figure 4 shows quantitative data characterizing these changes in the form of temperature dependencies of high-field susceptibility χ in films of different compositions. The value of χ was determined from the slope of the magnetization curves in fields above 40 kOe, when the change in M became practically linear. Along with the already noted decrease in the value of χ (T = 5 K) in binary $Gd_{100-x}Co_x$ films, attention is drawn to the peculiar course of the dependencies $\chi(T)$, which systematically changes with increasing x. With the absence or low content of Co on the curves $\chi(T)$, there is a more or less pronounced maximum in the region of the

Curie temperature of the crystalline phase and a rise at low temperatures, where, in our opinion, a set of Curie temperatures T_{Ci} is localized (see point 2). With increasing x, these features shift toward the middle of the temperature range, forming a single maximum near the Curie temperature of the amorphous phase. The presented data allow us to conclude that the speromagnetism of the system, which within the framework of the presented ideology is characterized by the value of χ, is closely related to the features of ferromagnetic ordering.

Some information about the transformation of magnetism in the Gd-Co system during its amorphization is also provided by the temperature dependencies of the coercive force $H_c(T)$ of films, shown in Figure 5.

Fig. 5. Temperature dependencies of the coercive force of $Gd_{100-x}Co_x$ films.

It should be kept in mind that H_c is a characteristic of the ferromagnetic state and is closely related to the magnetic anisotropy of the medium. Thus, the presence of magnetic hysteresis shows that in the crystalline state, Gd films exhibit magnetic anisotropy, although by the standards of rare-earth magnets, it is small. The latter follows from the relatively low level of coercive force (up to 600 Oe). The sharp downward trend of the curves $H_c(T)$ in films with low Co content can be interpreted as evidence of a rather strong temperature dependence of this anisotropy. The transition to an amorphous state, observed with increasing Co content, leads to a multiple decrease in coercive force, which is a natural consequence of the destruction of crystalline magnetic anisotropy.

CONCLUSION

A certain volume of experimental data has been obtained, allowing for a detailed analysis of changes in the magnetic properties of Gd-Co system films with a relatively low Co content (less than 20 at.%). It is shown that the films are heterogeneous in structure, which is represented as a superposition of crystalline HCP and FCC phases, as well as an X-ray amorphous phase. As the Co content increases, the structural dispersion deepens in both crystalline and, apparently, X-ray amorphous components.

The heterogeneity of the atomic structure is reflected in certain specifics of the magnetic state of the films. It is interpreted as a superposition of ferromagnetic and speromagnetic states associated with different structural phases. Moreover, in pure Gd films, ferromagnetism is inherent in the crystalline and, probably, nanocrystalline components. It is assumed that the latter is part of the X-ray amorphous phase and is characterized by the dependence of the Curie temperature on the size of nanocrystallites.

The deepened amorphization that occurs with increasing Co concentration, on the one hand, destroys the ferromagnetism and magnetic anisotropy of the crystalline state, but on the other hand, eliminates the exchange frustration of the atomic magnetic moments of Gd. As a result, a compromise ferromagnetic structure forms throughout the material, but with a reduced Curie temperature (compared to the crystalline state).

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CONFLICT OF INTERESTS

The authors of this work declare that they have no conflict of interest.

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