THE EFFECT OF Ag ADDITIVES ON THE STRUCTURE OF Fe-Pt FILMS

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Homogeneous thin films of FePt and Fe-(Pt,Ag) were obtained by modernized three-electrode ion-plasma sputtering. The film thickness was 220–450 nm. In this case, the calculated cooling rate reached ~10^{12}–10^{14} K/s. The structure of films was studied by X-ray diffraction and electron microscopy. It was found that nanocrystalline and amorphous phases were formed in freshly deposited films. It was shown that the resulting metastable structures were stable when heated to 670–890 K, depending on the composition. It was found that Ag additives reduced the coercive force of the films. It was shown that heat treatment increased the coercive force up to 36 kA/m in FePt films and up to 10 kA/m in Fe-(Pt,Ag) films. The conditions for obtaining films with low values of the temperature coefficient of electrical resistance (~$1.1\cdot10^{-5}$ K^{-1}) were determined.

Keywords: thin film, ion-plasma sputtering, coercive force, metastable state, amorphous and crystalline phases.

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1. Introduction

The refinement of the domain structure, the creation of thin diamagnetic layers between domains is one of the ways to increase the coercive force and magnetic energy of materials. Attempts to control the structure in such a way in thin film materials are known from the literature [1, 2]. In addition, the actual two-dimensionality of the films opens the possibility of their use in the manufacture of miniature magnets, rotors of any configuration for miniature micromotors or for the development of all kinds of magnetic sensors.

Potentially attractive alloys with improved magnetic properties include thin-film alloys based on components with limited mutual solubility in the liquid state. Such systems are characterized by a high surface energy of crystals [3-6], a large difference in the density of components, and a high positive heat of mixing of the alloy components. This makes it difficult to obtain alloys based on such components. Non-equilibrium conditions for obtaining a material make it possible to overcome the influence of the positive heat of mixing and obtain a new class of materials based on immiscible components. To date, quite a lot of single-phase alloys have already been obtained in such systems by various methods [7-14]. In the case of ion-plasma sputtering of atoms, the effective energy relaxation rate, according to theoretical estimates, reaches 10^{12}-10^{14} K/s [15]. This opens the possibility of the appearance of nonequilibrium phases during ion-plasma sputtering.

Homogeneous structures based on FePt and Fe-(Pt,Ag) alloys were obtained by the method of modernized three-electrode ion-plasma sputtering (MTIPS). These alloys are very promising since they have a high energy of uniaxial anisotropy and a high coercive force. Interest in the study of compounds of these alloys with the addition of diamagnetic Ag is also caused by the fact that such a compound can combine the properties of magnetically soft and magnetically hard materials and opens the possibility of a wide variety of applications.

2. Experimental procedure

The objects of study were films of alloys of the following compositions (at.%): Fe-11%Pt (D~450 nm); Fe-21%Pt (D~430 nm); Fe-39%Pt (D~320 nm); Fe-11%Pt alloy doped with 11%Ag diamagnetic additive (Fe-22%(Pt/Ag); Fe-31%(Pt/Ag); Fe-39 %Pt, in which some part of the paramagnet (Pt) was replaced by a diamagnet (Ag) and a film of Fe-39% (Pt/Ag) – $Fe_{61}Ag_{20}Pt_{19}$ was obtained.

The films were deposited simultaneously on glass-ceramic substrates and on fresh cleavages of NaCl single crystals. by the method of modernized three-electrode ion-plasma sputtering (MTIPS) of composite targets [5,7,8].

The composition of the films was chosen considering information about the concentration range of the existence of equilibrium phases in Fe-Pt, Fe-Ag, Ag-Pt alloys [16]. The target consisted of 16 cells in the form of parallelepipeds separated from each other by barriers with the function of electrostatic lenses. The parallelepipeds were made from pure elements (not less than 99.99%). The thickness of the obtained films was 110–450 nm.

The content of elements in the films was estimated with an accuracy of 0.5% (at.) using a technique that considers the relationship between the relative area of the substrate occupied by the element and its content in the deposited film [8]. After separation from the salt, the phase composition of the films in the as deposited and heat-treated states was studied. We applied the methods of electron microscopy using the device EMMA-2U and X-ray diffraction analysis using the device URS-2.0. The physical properties and thermal stability were studied on films deposited on glass-ceramic substrates. The electrical resistance of the films was measured by the four-probe method during continuous heating in a vacuum of ~10 mPa at controlled heating rates from 4 to 18 K/min. The magnetic properties were studied using a vibrating magnetometer in a maximum magnetizing field of 0.3 T, oriented parallel and perpendicular to the film surface.

3. Results and discussion

According to the equilibrium diagram, in the Fe-21%Pt alloy, the ordered Fe_3Pt (γ_1) phase at 1013 K should form, in the Fe-39%Pt alloy, the FePt (γ_2) phase at 1573 K, with the following homogeneity regions: γ_1 - 19 -33 at. %Pt, γ_2 - 35-59 at. %Pt.

In as-sputtered Fe + 11 at. % Pt films, a nanocrystalline phase with a coherent scattering region of about 3.4 nm in size was observed. After heat treatment (heating and cooling in vacuum) to the temperature of 903 K, the nanocrystalline phase passes into the metastable fcc FePt phase with lattice period a=0.3724 nm [17].

In as-sputtered film of composition Fe + 21 at. % Pt, the formation of amorphous phase with a CSR size of L≈1,6 nm was observed. After heating to the temperature of 903 K and cooling, metastable fcc FePt solid solutions are formed with a period a=0.3760 nm.

A dispersed (Fig. 1) solid solution of FePt is formed in Fe+39% Pt films in as-deposited state. After heating to the temperature of 903 K and cooling, coarsening of the structure is observed, and the lattice period of the FePt solid solution practically does not change. The formation of the Kurnakov phase in the studied films is not fixed.

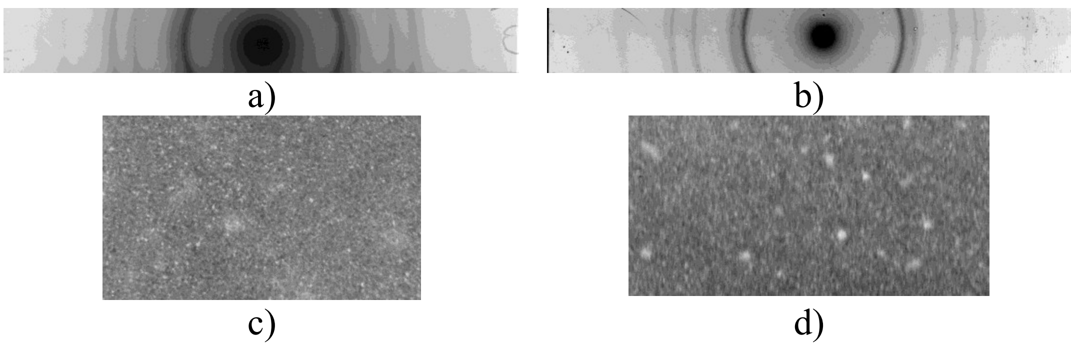

Fig. 1. Results of electron diffraction studies of the Fe-39%Pt film: a), c) ×37000 as-sputtered state and b), d) ×30000 after heating to 823 K..

The analysis of the obtained radiographs and the calculated sizes of coherent scattering regions (CSR) shows the following:

1) in the as-sputtered state, in all deposition modes, a nanocrystalline phase (NCP) is formed with the sizes of the coherent scattering regions (CSR) L ~ 3.0 – 3.5 nm;

2) after heating to 825 K, traces of NCP decay appear (a second blurred peak and a subpeak on the main halo from the side of large angles), but no noticeable increase in CSR is observed (L ~ 4.5 – 5.0 nm). In this case, the weak growth of OKR may indicate the emergence of internal tensions that arise because of the beginning of the collapse of the NCP.

The limits of thermal stability of the resulting metastable structures are established. The thermal resistance to structural transformations in FePt alloys decreases from 890 K in Fe+11 at. % Pt films to 680 K in Fe+39 at. % Pt films.

The temperature coefficient of resistance (TCR) of FePt films in a freshly deposited state varies from $6.5 \cdot 10^{-4}$ to $18.8 \cdot 10^{-4}$ K^{-1} depending on the content of Pt. Additions of Ag reduce the temperature of the beginning of changes in the structure of Fe-(Pt/Ag) films to 670-660 K, TCR of Fe-22…39% (Pt/Ag) films decreases to precision values of $1.1 \cdot 10^{-5}$ K^{-1} in the initial state.

Fe-Pt films are characterized by anisotropy of magnetic properties. At a perpendicular orientation of the magnetic field, the films exhibit weak hysteresis properties. For freshly deposited Fe-Pt films, with increasing Pt content, the coercive force increases from 5 kA/m to 7 kA/m. After heating the films in vacuum to 770–780 K, the coercive force increases by more than 10 times, reaching 36 kA/m in Fe-39%Pt films. In Fe-22…36%(Pt/Ag) films after heating up to 740 K the coercive force reaches 8.5 kA/m…12 kA/m. The improvement of the magnetic characteristics can be realized by choosing the holding time at a given temperature.

Conclusions

It has been shown that the addition of Ag to FePt alloys during modernized three-electrode ion-plasma sputtering leads to the formation of homogeneous thin films in the nanocrystalline state. After heating to 900 K, internal stresses arise in the films and the beginning of NCF decomposition is observed. In the initial state of the Fe-Pt film; Fe-(Pt/Ag) are magnetically soft material. After heating to 780 K, the coercive force of the film increases to 12 kA/m. The film becomes a hard magnetic material. The heat treatment regimens and compositions with a low value of the temperature coefficient of resistance (TCR ~ $1.1\ 10^{-5}$ K^{-1}) are determined. The studied films are promising from the point of view of developing miniature magnets and devices for high-density magnetic recording of information.

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