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The Physical Properties of the FeBiMo Films, Obtained by Ion-Plasma Sputtering

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The paper presents the results of investigations of the phase composition and physical properties of the pure Bi, Mo and FeBiMo films, with thickness d~100-500 nm, obtained by three-electrode ion-plasma sputtering. X-Ray analysis showed that in the as-deposited Bi films is formed a mixture of rhombohedral and cubic nanocrystalline Bi phases and traces of Bi2O3 oxide. Heat treatment at 650 K leads to the decay of the Bi non-equilibrium phase. In the Mo original films is formed a mixture of Mo cubic phase and MoO2, MoO3 oxides. As a result of heat treatment at the temperature of about 870 K phase composition and the grain size remains unchanged. FeBiMo films in the initial state is a mixture of rhombohedral and cubic Bi phases and the hexagonal Fe phase. In the Mo-doped films non-equilibrium Bi phase remains after heat treatment at 770 K. Construction of the temperature dependence of resistivity has revealed the reversible phase transition in the Bi films. A similar behavior of Bi properties is also manifested in FeBiMo films. The analysis of demagnetization curves of Bi films showed a manifestation of hysteresis properties, which is not typical for diamagnetic. This can be explained by the Bi2O3 phase presence.

Keywords: FeBiMo Films, Ion-Plasma Sputtering, Non-Equilibrium Phase, Reversible Phase Transition, Ferrimagnets, Nanocrystalline Phase.

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1. INTRODUCTION

Currently magnetoelectric materials are widely used in sensor technology, spintronics, as well as storage devices of the information. Bismuth ferrite is a well studied magnetoelectrics with sufficiently high phase transition temperatures and has a magnetoelectric effect at room temperature, which leads to its potential for application in spintronics.

In bulk bismuth ferrite there is a problem of charge leakage due to non-stoichiometry and defects that limit the practical application of this material as a bulk crystal. In the Bi_2O_3-Fe_2O_3 system easily generated non-equilibrium states due to the proximity of phase transition temperatures. Production of thin films allows change the bismuth ferrite properties in comparison with properties in a bulk form.

The aim of our work was to obtain a similar film material doped with refractory component in order to improve thermal stability of the films, obtained by three-electrode ion-plasma sputtering. In this method ultra-rapid cooling rate allows to obtain such non-equilibrium structures [1].

The effective cooling rate of films depends on the relaxation time of individual atom on a substrate and its estimated value is 10^{12} - 10^{14} K/s [2]. Thus, we can speak of a “quenching from the vaporous state” (QVS).

2. MATERIALS AND METHODS

2.1 Objects of investigations

The objects of investigations were pure Bi, Mo and FeBiMo films. Film thickness are d ~ 100-500 nm, estimated by weighing the substrates before and after sputtering. Sputtering was carried out on Na-Cl single-crystal substrates for the structural investigations by X-Ray analysis. The films obtained under identical deposition conditions on the sitall substrate were used for studying the thermal stability and physical properties of non-equilibrium structures.

2.2 Methods of investigations

For the films obtaining used the method of ion-plasma sputtering in vacuum. Type-setting of targets placed directly on the sputtering surface of the component squares. An additional acceleration of Ar ions which was impinging on the target and increase in the kinetic energy of the depositing onto sitall substrates atoms was achieved by using a specially designed device in the sputtering setup URM 3.279.014 [3].

The surface resistance was measured by the four point technique upon continuously films heating in the vacuum 10 mPa. The structure and compositions of the initial and annealed films were studied by the X-Ray diffraction obtained with Debye camera with CoK_{α} radiation. The coercive force Hc was measured on a vibration magnetometer in the magnetizing field 0.3 T with parallel and perpendicular orientations.

Calculation of the activation energy was carried out by Kissinger method [4]. Using the temperature dependence of the resistance at different heating rates you can construct the function ln(T2/V) from F(1000/T). This relationship satisfies the Arrhenius equation. The phase transitions activation energy was estimated by the slope of this line.

The films vapor deposition conditions shown in the Table.1.

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Table 1 – Conditions of sputter deposited films

	Bi*	Bi**	Mo*	Mo**	Fe – 18 at. %, Bi – 18 at. %, Mo
U, kV	2	2	2	2	2
I_A, A	0.8	1	0.8	2	2
P_{Ar}, mPa	16	120	16	53	53
d, nm	500	500	100	160	260

Where *U* - target voltage; I_A - anode current; P_{Ar} - pressure of orifice gas (Ar); *d* – film thickness

3. RESULTS AND DISCUSSION

The X-Ray analysis and estimation of the coherent-scattering regions size L (CSR) showed that in the as-deposited Bi films is formed the mixture of nanocrystalline rhombohedral (L ~ 6.5 nm) and cubic Bi phases and traces of Bi_2O_3 oxide. Heat treatment of pure Bi films in a vacuum at the temperature of about 650 K leads to non-equilibrium Bi phase disappearance and rhombohedral Bi grain growth (L ~ 8 nm) (Fig. 1).

In the original Mo films, obtained under conditions of lower anode current and working gas pressure (Mo*), is formed a mixture of nanocrystalline Mo phase (L ~ 3.6 nm) and MoO_3 oxide. When changing the deposition conditions ib the original Mo films (Mo**) is formed a MoO_2 oxide (L ~ 3.4 nm) as well as phase traces described above. The films structure and phase composition did not change after heat treatment at the temperature of 870 K.

FeBiMo films in the initial state is a mixture of rhombohedral Bi (L ~ 5.6 nm), cubic Bi and hexagonal Fe phases. The grain growth of rhombohedral Bi occurs after heat treatment (L ~ 7 nm). Non-equilibrium Bi phase is observed even at high temperature treatment (~ 770 K) that is caused Mo addition.

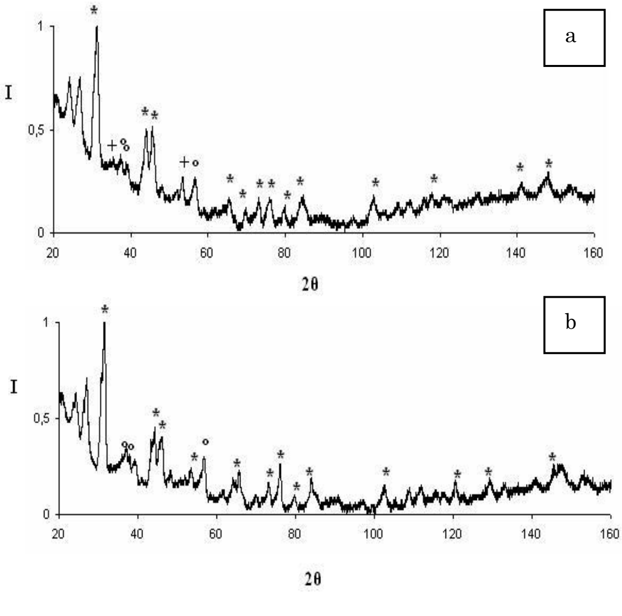

Fig. 1 – X-ray diffraction patterns from pure Bi films freshly deposited (a), after heat treatment (b), * - rhombohedral Bi phase; + - cubic Bi phase; ° - Bi_2O_3 oxide

Investigation of the temperature dependence of resistivity showed that heat curves are characterized by three main sections for all films. In the first section there is a smooth decrease in resistivity with temperature increasing. On the second segment there is a rapid decrease of the resistance in a relatively short temperature range. This is a result of phase transitions or recrystallization processes. The third section is characterized by a gradual increase in resistivity (Fig. 2).

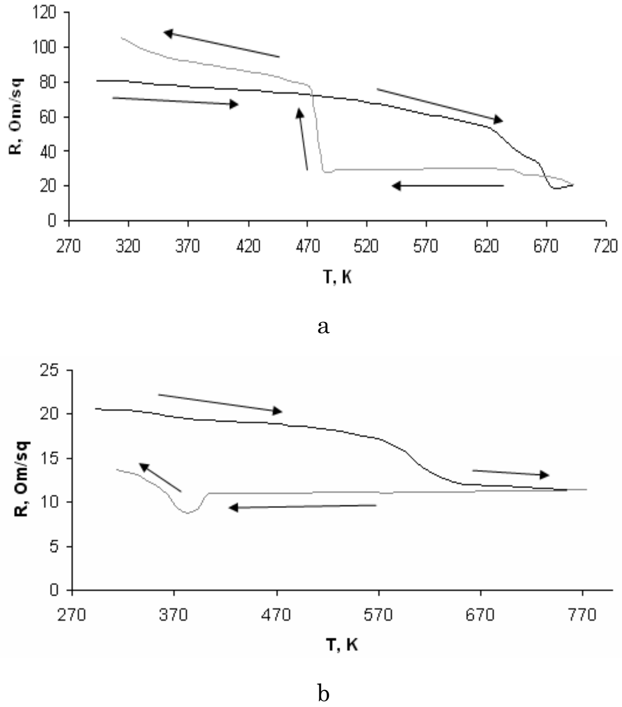

Fig. 2 – Temperature variation of resistivity of the Bi (a) and FeBiMo (b) films

Cooling of the Bi containing films leads to a specific behavior of the resistivity. In the first phase it remained virtually unchanged. When reaching the certain temperature the resistance increases abruptly, after which continues relatively smooth growth.

It should be noted that for the pure Bi films reverse phase transition temperature under cooling is about 470 K, while for the FeBiMo films this temperature is about 370 K. This temperature difference is probably related with the influence of Mo refractory.

The analysis of the phase transition temperature shift with an increase in the heating rate allowed for the calculation of the phase transition activation energy by Kissinger method.

For pure Bi films activation energy is EA ~ 8500 K. The activation energy of the Mo films is in the range of EA ~ 6500-9500 K depending on the deposition conditions. FeBiMo films investigations of the temperature dependence of resistivity showed the highest value of energy EA ~ 11000 K.

The analysis of the demagnetization curves of Bi films showed a manifestation of hysteresis properties. Since pure Bi is a diamagnetic, we can assume that this behavior is due to the presence of Bi_2O_3 and non-equilibrium Bi phases. The magnetic moment is uncompensated which is characteristic of ferrimanetic.

4. CONCLUSIONS

The phase composition and physical properties of the pure Bi, Mo and FeBiMo films obtained by three-electrode ion-plasma sputtering method were studied by X-Ray analysis, measuring of relative electrical resistance and magnetometric measuring.

As a result, it should be noted that the selection of deposition conditions allows to obtain films with different oxide groups, which is an important task in the multiferroic research field.

Adding to the films with non-equilibrium structures Mo refractory helps to prevent the decay of non-equilibrium phases and thus increase the thermal stability of the film material.

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