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METHODS OF MATERIALS PROPERTIES ANALYSIS

Phase Composition, Structure, and Magnetic Properties of Fe–Zr–N Films Produced by Magnetron Sputtering of Heated Target

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Abstract—The phase-structural state and magnetic properties, specifically, the saturation induction B_s and the coercive field H_c , of Fe–Zr–N films are characterized in a wide range of chemical compositions. The films are obtained by reactive magnetron sputtering in an Ar + N₂ atmosphere at different concentrations of N₂ (0, 5, and 15 vol %) using the FeZr_X targets heated above the Curie temperature. Single-phase (bcc α -Fe(Zr,N) solid solution), two-phase (α -Fe(Zr,N) + Fe₄N, α -Fe + ZrO_{2-x}), and three-phase (α -Fe(Zr,N) + Fe₄N + Fe₃N) films with a nanocrystalline (grain size of 2 to 14 nm), amorphous, and mixed (amorphous + nanocrystalline) structure are formed. The nanocrystalline films are strong ferromagnets exhibiting a high saturation induction B_s (0.5 to 2.1 T) and low coercive field H_c (40 to 1100 A/m). The films with the amorphous and mixed (amorphous + nanocrystalline) structure are weak ferromagnets (B_s is about 1 to 7 mT).

Keywords: Fe-based soft magnetic films, nanocrystalline, magnetron sputtering, phase-structural state, static magnetic properties

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INTRODUCTION

The revolutionary development of nanotechnologies in the direction of broadening the spectrum of functional properties of magnetic electronics and creation of micro- and nanosized devices [1] places high demands on the set of properties of soft magnetic materials, in particular, the still not achieved combination of a high saturation induction $(B_s \ge 2 \text{ T})$ and minimum possible coercive field ($H_c < 80$ A/m). In this connection, the class of nanocrystalline films on the basis of $Fe-M_{IV}X$ alloys is of considerable interest [2-4]. A two-phase structure (nanocomposite) comprising a soft magnetic phase with the inclusions of a hard thermodynamically stable nonmagnetic $M_{IV}X$ phase (nitrides, carbides, and borides of metals belonging to the IVA group of the periodic system) can be formed in films obtained by the method of magnetron sputtering with subsequent annealing. The films with such a structure can afford the combination of a high B_s value with a low H_c value. Despite the fundamental significance for the creation of soft magnetic materials with specified properties, studies on the relationship between the chemical composition and the preparation and thermal treatment conditions of such class of films and their phase-structural and magnetic properties have hardly been reported in the literature.

The aim of the present study is investigation of the relationship between the chemical composition and annealing temperature of the Fe–Zr–N films obtained by magnetic sputtering of a heated target and their phase-structural state and magnetic properties.

EXPERIMENTAL

The films for investigations containing about 3 at % Zr (hypoeutectic composition on the Fe–Zr phase diagram [5]), about 9 at % Zr (composition close to the eutectic), and about 33 at % Zr (hypereutectic composition) with the Zr/N ratio in the range from 0.3 to 36.5 at % depending on the Zr content were obtained using $FeZr_x$ targets made of Fe disks with uniformly distributed Zr pieces. The surface melting of Fe and Zr with the formation of an alloy in the erosion zone took place in the magnetron chamber under the influence of a plasma torch. The target with the produced erosion zone of relevant composition was heated above the Curie temperature before each sputtering run, which made the sputtering process possible. The deposition was carried out in a gaseous atmosphere of Ar or $Ar + N_2$ with the volume fractions of N_2 of 0, 5 and 15% in the gas mixture. Plates of glass and Ni-Cr alloy were used as substrates. The prepared films were annealed for 1 h in vacuum ($p \sim 10^{-4}$ Pa) at

| Film series (*) | Chemical composition, at % | | | | Phase composition vol % of phase | Lattice | Grain size, D, |
|--------------------|----------------------------|------|------|------|------------------------------------|-------------|----------------|
| | Fe | Zr | Ν | 0 | Thase composition, vor // or phase | of phase, Å | nm |
| I (0) | 81.1 | 3.2 | <2 | 13.7 | bcc α-Fe [65] | 2.868 | 28.4 |
| | | | | | fcc ZrO_{2-x} [35] | 5.019 | 2.9 |
| II (5) | 90.4 | 2.9 | 4.7 | 2.0 | bcc α-Fe(Zr,N) [~100] | 2.904 | 13.6 |
| III (5) | 75.8 | 8.7 | 6.8 | 8.7 | bcc α-Fe(Zr,N) [~100] | 2.900 | 3.5 |
| IV (15) | 82.5 | 3.1 | 6.4 | 8.0 | bcc α -Fe(Zr,N) [58] | 2.884 | 7.5 |
| | | | | | bcc Fe ₄ N [42] | 3.831 | 8.1 |
| V(15) | 86.0 | 4.3 | 6.7 | 2.9 | bcc α -Fe(Zr,N) [58] | 2.918 | 2.0 |
| | | | | | bcc Fe ₄ N [42] | 3.798 | 5.5 |
| VI (15) | 87.3 | 2.4 | 7.4 | 2.9 | bcc α -Fe(Zr,N) [31] | 2.886 | 4.5 |
| | | | | | bcc Fe ₄ N [64] | 3.831 | 7.6 |
| | | | | | Fe_3N [5] | 4.686/4.301 | 9.1 |
| VII (15) | 43.3 | 34.8 | 12.4 | 9.6 | XRD amorphous [~50] | _ | _ |
| | | | | | fcc ZrN [~50] | 4.585 | 5.3 |
| VIII (0) | 61.9 | 32.9 | <1 | 4.3 | XRD amorphous [~100] | _ | — |

 Table 1. Results of the chemical and phase analysis of the films in the initial state

*The N_2 content in the gaseous atmosphere, vol %.

300, 400, 500, and 600° C, i.e., in the temperature range in which a nanocomposite structure can be formed according to [3].

The elemental compositions of the films deposited on the glass substrates were determined by the method of energy dispersive X-ray spectroscopy. The method of glow discharge optical emission spectroscopy was used for a more accurate determination of light elements (O and N). The depth distribution profile of elements deposited on the model substrates of Ni-Cr alloy disks and the film thicknesses, which ranged from 1 to $1.8 \,\mu\text{m}$, were determined by this method. The phase-structural state was studied by the X-ray diffraction method (the Rietveld full-profile method). The preliminary data treatment and the qualitative and quantitative phase analysis were carried out using the OUTSET, PHAN, and PHAN% software packages [6]. The lattice parameters of phases and the grain size, which was taken equal to the coherent scattering cross section, were determined. The magnetic hysteresis loops were measured on a LakeShore 7407 vibration magnetometer (USA) with an error of about 10%in determination of the saturation induction. All the measurements were carried out at room temperature.

RESULTS AND DISCUSSION

The chemical analysis of the investigated films shows that the following elements are present in all of them: Fe, Zr, N, and O (Table 1). The presence of oxygen in the films might be related to an insufficient level of evacuation of the vacuum chamber (the residual pressure is about 10^{-3} Pa) and also to its contamina-

tion in the working gas atmosphere (the purity of Ar is 99.993%, and the purity of N is 99.999%).

A single-phase nanocrystalline structure of the α -Fe(Zr,N) ferromagnetic phase, which consists of a solid solution of N and Zr in α -Fe having the bcc crystal lattice with a grain size of 3.5 to 13.6 nm depending on the chemical composition (Fig. 1, Table 1), is formed in the films containing about 3 and 9 at % Zr and obtained in a gaseous atmosphere with 5 vol % N₂ (series II and III). The formation of a solid solution is confirmed by the parameter of the bcc phase lattice exceeding a tabulated value of 2.866 Å for pure α -Fe (Table 1). An increase in the nitrogen content of the gas mixture from 5 to 15 vol % (series IV–VI) during the preparation of these films gives rise to the formation of a two- and three-phase structure; i.e., Fe₄N and Fe₃N nitrides are formed in addition to the bcc phase of α -Fe (Zr.N). The grain size of the bcc phase is decreased from 13.6 nm (single-phase) to 2.0 nm (for two- and three-phase films) with an increase in the total content of Zr and N in the film and in the solid solution on the basis of α -Fe, respective (Table 1, Fig. 2).

The observed pattern of behavior of the grain size of the bcc phase indicates the influence of the solid-solution (Zr and N) and dispersion (Fe₃N and Fe₄N) hardening of the bcc phase, retarding the growth of grains of this phase in the process of film formation.

The films with a content of about 3 at % Zr produced in the Ar gas atmosphere (series I) are characterized by a high concentration of oxygen (13.7 at %) (Table 1). The largest oxygen content among the investigated films is connected with the fact that they



Fig. 1. X-ray diffractograms of films in the initial state.



Fig. 2. Dependence of the grain size of the bcc phase on the basis of α -Fe on the summarized content of Zr and N in the initial films.

are obtained at the largest pressure of the gas mixture in the magnetron, at the maximum voltage on the cathode, and at the highest starting temperature of the substrate in comparison with the other investigated films (1 Pa, 500 V, and 160°C vs. ~0.15 Pa, ~450 V, and ~80°C). A two-phase nanocrystalline structure is formed in these films represented by the bcc phase on the basis of α -Fe, slightly alloyed by Zr, and N with a grain size of about 30 nm, and the ZrO_{2-x} oxide phase of a nonstoichiometric composition in the fcc modification with a grain size of about 3 nm formed in an amount of 35 vol % (Table 1).

The formation of the phase of α -Fe, slightly alloyed by Zr and N is proved by the proximity of the lattice constant values of the bcc phase (2.868 Å, Table 1) and of pure α -Fe. The formation of the oxide phase of a nonstoichiometric composition is proved by the lattice constant of this phase (5.019 Å, Table 1), which is much smaller than the lattice constant of the γ -ZrO₂ phase in the area of its homogeneity (5.09 to 5.27 Å) [5]. In the conditions of the absence of solubility in α -Fe, on one hand [5], and of the highest affinity to Zr (the standard formation enthalpy $-\Delta H_{\rm f}^0 = 130$ kcal/(mol atom) [7]) among the all incorporated phases produced in the Fe–Zr–N–O system, on the other hand, oxygen reacts with Zr with the formation of a ZrO_{2-x} phase during the formation of these films. The large grain size of the ferromagnetic bcc phase (about 30 nm) in these films indicates the absence of the effect of the solid-solution and dispersion hardening (ZrO_{2-x}) phase).

An amorphous (XRD amorphous) phase is formed in the films with a high Zr content (series VII and VIII) in the initial state owing to the amorphization influence of Zr (Fig. 1, Table 1). In addition to the amorphous phase, nitride ZrN of the fcc modification with a grain size of 5 nm is formed in the films of this group with an increase in the N content from 0.9 to 12.4 at % (Table 1).

The formation of the Fe₄N and Fe₄N/Fe₃N phases in the films of series IV, V, and VI, the ZrN phase in the films of series VII, and the ZrO_{2-x} phase in the films of series I indicates the competition of the of thermodynamic and kinetic factors during the condensation and growth of films of different compositions. Since the affinity of Zr to N ($-\Delta H_f^0 = 355 \text{ kJ/(mol atom)}$) is significantly larger than the affinity of N to Fe ($-\Delta H_f^0 =$

Significantly larger than the animity of N to Fe ($-\Delta H_f = 21 \text{ kJ/(mol atom) [7]}$), the formation of the ZrN phase should be expected from the point of view of thermodynamics in all the investigated films except the films of series I, in which N is not introduced. The appearance of the Fe₄N and Fe₄N/Fe₃N phases in the films of series IV, V, and VI indicates the prevalence of the kinetic factor upon the formation of the phase composition of these films. It is determined by the time of diffusion of the N atoms to the Fe atoms surrounding them in a large amount, which is significantly smaller than the time of diffusion of the N atoms to the Zr atoms (mass action law). The thermodynamic factor determines the formation of the ZrO_{2-x} phase ($-\Delta H_f^0 =$ 544 kJ/(mol atom) [7]) in the films of series I.

Annealing of the films containing about 3 and 9 at % Zr (series II–VI) does not change the phase composi-



Fig. 3. Influence of the annealing temperature (a) on the crystal lattice parameter and (b) on the grain size of the bcc phase on the basis of α -Fe in the investigated films.

tion formed in the initial state, but leads to depletion of the bcc solid solution on the basis of α -Fe by alloying components (Zr and N), which proceeds to a greater extent the higher the annealing temperature, and also contributes to some increase in the volume fraction of the Fe₄N and Fe₃N phases. A decrease in the lattice constant of the bcc phase indicates the depletion of the solid solution by alloying elements with an increase in the annealing temperature (Fig. 3a). The absence of the growth of grains of the bcc phase in practically all the films of this group with an increase in the annealing temperature, at least up to 500°C (Fig. 3b), indicates the influence of the effect of solid-solution and dispersion hardening of the bcc phase. It should be noted that the main contribution to the thermal stability of the ferromagnetic grain size, i.e., to hardening of the material, is made by solid-solution hardening. This is confirmed by the fact that the bcc phase lattice parameter after annealing at 600°C decreases from the films of series II to the films of series III, and then to the films of series V (Fig. 3a). At the same time, the grain size of the bcc phase increases with respect to the unannealed films by about 1.5, 2, and 7 times, respectively (Fig. 3b, Table 2).

Annealing of the films with a high Zr content (series VIII) at 500°C gives rise to the partial crystalli-



Fig. 4. Influence of the annealing temperature on the (a) saturation induction and (b) coercive field values in the investigated films.

zation of the amorphous phase with the formation of about 50 vol % ZrO_2 with a grain size of 16 nm, which increases to about 23 nm after annealing at 600°C (Table 2). Annealing of the films of series VII at 400°C does not change the following phase state formed upon sputtering: about 50 vol % of the amorphous phase and about 50 vol % of the fcc ZrN phase (Table 2). Thus, an amorphous phase stable to a large extent up to 600°C is formed in the films with a high Zr content (about 33 vol %).

On the basis of the results of measurements of the magnetic properties, the investigated films can be divided into the following two groups: strong ferromagnets (series I–VI) with a high value of the saturation induction B_s (0.5–2.1 T) and a low value of the coercive field H_c (40–1100 A/m) (Fig. 4) and weak ferromagnets (series VII and VIII) with $B_s = 1-7$ mT and $H_c =$ 16–4000 A/m (Fig. 4a). The value of B_s in the first group of films is determined by the following four factors: (1) the volume fraction of the ferromagnetic phase on the basis of α -Fe; (2) the degree of its alloying with N and Zr (estimated by crystal lattice parameter of the bcc phase); (3) the volume fraction of the ferromagnetic nitride phase (Fe_4N and Fe_3N) or the nonferromagnetic oxide phase (ZrO_{2-x}) ; and (4) the Fe content in the film. Thus, the value of B_s is

| Film | Phase composition, vol % (grain size after annealing, nm) | | | | | | | |
|--------|-----------------------------------------------------------|--------------------|-------------------------------|-------------------------------------------------|-------------------------------------|--|--|--|
| series | initial | 300°C | 400°C | 500°C | 600°C | | | |
| II | α-Fe(Zr,N) | α-Fe(Zr,N) | α -Fe(Zr,N) | α-Fe(Zr,N) | α-Fe(Zr,N) | | | |
| | [~100] (13.6) | [~100] (14.0) | [~100] (14.8) | [~ 100] (18.4) | [~100] (21.4) | | | |
| III | α-Fe(Zr,N) | α -Fe(Zr,N) | α -Fe(Zr,N) | α -Fe(Zr,N) | α -Fe(Zr,N) | | | |
| | [~100] (3.5) | [~100] (4.1) | [~100] (3.8) | [~100] (4.0) | [~100] (6.9) | | | |
| IV | α-Fe(Zr,N) [58] (7.5) | * | α-Fe(Zr,N) [53] (8.2) | α-Fe(Zr,N) [48] (8.6) | * | | | |
| | Fe ₄ N [42] (8.1) | | Fe ₄ N [47] (8.3) | Fe ₄ N [52] (8.5) | | | | |
| V | α-Fe(Zr,N) [58] (2.0) | * | α-Fe(Zr,N) [61](2.0) | α-Fe(Zr,N) [51] (2.9) | α-Fe [28] (14.6) | | | |
| | Fe ₄ N [42] (5.5) | | Fe ₄ N [39] (6.7) | Fe ₄ N [49] (6.1) | Fe ₃ N [57] (3.4) | | | |
| | | | | | ZrO ₂ [15] (3.7) | | | |
| VI | α-Fe(Zr,N) [31] (4.5) | * | α -Fe(Zr,N) [34] (2.3) | * | * | | | |
| | Fe ₄ N [64] (7.6) | | Fe ₄ N [56] (10.2) | | | | | |
| | Fe ₃ N [5] (9.1) | | Fe ₃ N [10] (11.3) | | | | | |
| VII | XRD amorphous $[\sim 50]$ | * | XRD amorphous [~50] | * | * | | | |
| | ZrN [~50] (5.2) | | ZrN [~50] (5.3) | | | | | |
| VIII | XRD amorphous | XRD amorphous | XRD amorphous | XRD amorphous | XRD amorphous | | | |
| | [~100] | [~100] | [~100] | $[\sim 50]$ ZrO ₂ $[\sim 50]$ (16.5) | [~50] ZrO ₂ [~50] (22.7) | | | |

Table 2. Influence of annealing on the phase composition of films

*No annealing.

decreased from about 1.1-2.1 T in the single-phase films (series II and III) to about 0.6-1.6 T in the films containing the nitride phase (series IV–VI, Fig. 4a). At the same time, a decrease in the Fe content in the single-phase films from 90.4 at % (Series II) to 75.8 at % (Series III) leads to a decrease in $B_{\rm e}$ from about 2.1 to 1.1 T, respectively. A decrease in the Fe content from 86.0–87.3 at % (series V and VI) to 82.5 at % (series IV) in the heterophase films with nitrides reduces $B_{\rm s}$ from about 1.6 to 0.6 T, respectively (Fig. 4a). As seen from Table 1 and Fig. 4a, a general trend toward a decrease in B_s with a decrease in the Fe content is observed in all of these films. Annealing of the films of series III–V at a temperature in the range from 300 to 400°C leads to a decrease in the value of B_s , which might be connected with a decrease in the energy of exchange interactions due to change in the value of residual macrostresses, as was shown in this kind of films [8], and has almost no effect on the value of B_s of the rest of the investigated films (Fig. 4a).

The coercive field in the first group of films in the initial state, i.e., strong ferromagnets (series I–VI), decreases upon a decrease in the grain size of the bcc phase (Figs. 2 and 4b), which is consistent with the model of random magnetic anisotropy [9]. A non-monotonic change in the H_c value with an increase in the annealing temperature takes place in all the investigated films. It should be noted that the films characterized by the highest B_s value (series II, III, and V) show the smallest coercive field H_c after annealing at 500°C (Fig. 4b). This phenomenon, in view of unchanging grain size of the bcc phase upon an increase in anneal-

ing temperature to 500°C (Fig. 3b) and upon a decrease in the crystal lattice parameter (Fig. 3a), is connected, as shown in [10] for the films with a similar composition, with changes in the magnetic structure, in particular, with a decrease in the energy of the effective local magnetic anisotropy, an increase in the effective exchange energy, and also with a decrease in the residual macroscopic stresses in the films to a minimum [8]. An increase in the coercive field after annealing at 600°C (Fig. 4b) might also be connected with the growth of grains of the bcc phase (Fig. 3b).

Annealing of the films of the second group, i.e., weak ferromagnets (series VII and VIII)), does not lead to a change in the phase-structural state up to 400°C, and such parameters of the magnetic properties as the saturation induction B_s and the coercive field H_c in the films of series VII stay at a level of 63 mT and 4000 A/m, respectively.

The films of series VIII, which are weak ferromagnets in the initial state ($B_s = 7 \text{ mT}$), become strong ferromagnets ($B_s = 138 \text{ mT}$) after annealing at 600°C. Apparently, this might be connected with the phase-structural changes occurring in the amorphous phase upon annealing at 600°C (in addition to the formation of the ZrO₂ phase (Table 2)) and with the change in the chemical composition of the formed phases.

CONCLUSIONS

Using the method of reactive magnetron sputtering of the FeZr_X targets heated above the Curie temperature in an atmosphere of Ar + N₂ (0, 5, or 15 vol % N₂),

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the films containing from 3 to 35 at % Zr at the ratio of Zr/N (at %/at %) ranging from 0.3 to 36.5 were obtained on the glass substrates. Depending on the chemical composition, the single-phase (bcc α -Fe(Zr,N) solid solution), two-phase (α -Fe(Zr,N) + Fe₄N and α -Fe + ZrO_{2-x} , and three-phase (α -(FeNZr) + Fe₄N + Fe_3N) nanocrystalline (with a grain size of 2 to 14 nm), amorphous, and amorphous-nanocrystalline structures are formed in the films. Annealing of the films in the temperature range from 300 to 600°C leads to depletion of the bcc solid solution in zirconium and nitrogen in the absence of the growth of grains of this phase, to a slight increase in the volume fraction of the Fe₄N and Fe₃N phases, and to partial nanocrystallization of the amorphous phase. The nanocrystalline films are strong ferromagnets with high values of the saturation induction $B_{\rm s}$ (0.5 to 2.1 T) and low values of the coercive field H_c (40 to 1100 A/m). The B_s and H_c values decrease upon an increase in the annealing temperature, and the H_c value reaches a minimum at 500°C. It is shown that the saturation induction of the nanocrystalline films is determined by the volume fraction of the ferromagnetic phase, its chemical composition, the volume fraction of the nitride (Fe₄N and Fe₃N) or oxide (ZrO_{2-x}) phases, and the content of Fe in the film. The films having the amorphous or amorphousnanocrystalline structure are weak ferromagnets with a low saturation induction value (1 to 7 mT).

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