



Magnetic properties of CoFeP films prepared by electroless deposition

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ABSTRACT

Structure and magnetization of CoFeP films prepared by the electroless deposition were systematically investigated by varying the bath composition and deposition parameters to optimize soft magnetic properties. The cobalt content in the CoFeP films varies from 40.4 to 94.9 wt% by controlling the bath composition. Increase of the metallic ratio $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}/(\text{CoSO}_4 \cdot 7\text{H}_2\text{O} + \text{FeSO}_4 \cdot 7\text{H}_2\text{O})$ affects the films' microstructure, which switches from amorphous to crystalline structure. The magnetic properties of CoFeP films reveal that the coercivity (H_c) values range from 80 up to 185 A/m and the saturation magnetization (M_s) from 82 to 580 emu/g depending on the bath composition, deposition parameters and heat-treatment conditions. Increase of M_s and remanent magnetization (M_r) as well as decrease of H_c are observed for the CoFeP films with bath pH, temperature and the metallic molar ratio increasing. It is also found that the H_c is enhanced with the increase of $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ concentration. CoFeP films showing good soft magnetic properties with coercivities less than 140 A/m and M_s close to 600 emu/g can be obtained in high pH bath and thereafter heat treatment. The deposit is found to be suitable as soft magnetic materials for core materials.

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1. Introduction

Soft magnetic material is a prerequisite to obtain magnetic storage devices with high areal recording density. With the advent of nanoelectric devices, low coercivity (H_c) and high saturation magnetic flux density become the requirements needed for the fabrication of these magnetic storage devices [1–4]. The manufacture and properties evaluation of Co/Fe-based alloys are attracting more and more interest because they exhibit excellent properties as soft magnetic materials with low H_c , very weak magnetostriction, high saturation magnetization (M_s), and large permeability at high frequencies [1–8], thus make them a critical element in various applications and devices. Co/Fe-based films with low coercivities and high M_s can be obtained by physical/chemical deposition processes [7,9–12]. Fu et al. [7] deposited FeCo films with a 5 nm Co underlayer by facing target sputtering and control the magnetic anisotropy in high-moment soft FeCo film. The results of Yoo and Osaka group [9–11] indicated that the magnetic properties of electrodeposited FeCoNi alloys are strongly dependent on the alloy composition and structure. The magnetic measurement revealed that the H_c values of electroplated CoPd alloys ranged from 84 up to 555 Oe and the magnetic saturation from 0 to 1.73 T by controlling the pH and $[\text{Co}^{2+}]/[\text{Pd}^{2+}]$ ratio in the bath [13].

Electroless deposition is a chemical method to obtain homogeneous films on various substrates and is applied in wide industrial fields. Electroless deposition of Co-based alloys has already been studied by several work groups [14–19]. The works of Rudnik and Gorgosz [18] showed that the stationary potential, the deposition rates and the plating efficiencies changes with the concentration of the additive. These results are, however, still insufficient for describing the magnetic properties of electroless Co-based alloys because of the lack of systematic investigation on the preparation conditions and deposit magnetic properties. In this work, we are dealing with the magnetic properties of the electroless CoFeP films from a sulfate stable bath. The influence of bath composition and parameters on the film structure, composition and magnetic properties are discussed.

2. Experimental

CoFeP alloys were deposited onto brass plates with a size of $15 \times 5 \text{ mm}^2$. Prior the electroless deposition, the substrates were treated with HCl and alcohol solutions. The films were deposited from a sulfate bath as listed in Table 1. The pH of the solution was adjusted using ammonia and measured by the electronic pH-meter Orion model 720A. The electroless deposition was initiated galvanically by using Al foil. The effect of bath composition and deposition process on the composition, structure and magnetic properties of films, was investigated by varying the

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metallic ratio of $\text{Fe}^{2+}/(\text{Fe}^{2+}+\text{Co}^{2+})$ in the baths, pH and temperature. The bath temperature was controlled at about 70 °C except if stated otherwise. Some samples were heat treated for 1 h at temperatures of 200 and 400 °C to investigate the effect of annealing on the film structure and magnetic property. Heat treatment was carried out in a vacuum furnace to prevent the oxidation and the sample was cooled to room temperature in the furnace itself.

The composition of the films was investigated using SEM (JEOL JSM-6700) combined with energy-dispersive spectrometry (EDS). Analysis of crystal phases in the deposits was performed with an X-ray diffractometer (XRD) (Siemens D5000) with $\text{CuK}\alpha$ radiation and a scanning range of 10–100°. Magnetic properties such as H_c and magnetic saturation of the deposited films were determined using a vibrating sample magnetometer (VSM, HH-50).

3. Results and discussion

Fig. 1 shows the dependence of film composition on the bath composition and deposition condition. It can be seen from

Table 1
Formulation of the electrolytes and deposition conditions.

Chemical compounds	Concentration (M)
$(\text{Na})_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$	0.25
$\text{C}_{12}\text{H}_{12}\text{O}_{11}$	0.0058
H_3BO_3	0.5
$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	0–0.2
$\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$	0–0.2
$\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$	0–0.5
T	60–90 °C
pH	9.5–11.5

Fig. 1(a) that an increase in the $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ concentration from 0.1 to 0.5 M results in the Co content in the film slightly increasing from 57.6 to 66.4 wt%, P from 1.3 to 6.5 wt%, while the Fe content decreasing from 41.1 to 27.1 wt%. Fig. 1(b) displays film composition variation with the ratio of FeSO_4 concentration. It is clear that iron content in the film is increased from 0 to 57.9 wt%, while cobalt content is decreased from 94.6 to 40.4 wt% and phosphorus from 5.4 to 1.7 wt% when the metallic ratio $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}/(\text{CoSO}_4 \cdot 7\text{H}_2\text{O}+\text{FeSO}_4 \cdot 7\text{H}_2\text{O})$ increases from 0 to 67%. However, the bath pH and temperature have little effect on the film composition: The contents of phosphorus, iron and cobalt are about 1.3%, 41.7% and 57%, respectively, as shown in Figs. 2(c) and (d). The composition variance with deposition condition can be understood. It is known NaH_2PO_2 acts as reductant in electroless bath. An increasing in the $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ concentration may lead to more electrons released due to the oxidation of H_2PO_2^- . These released electrons would be gained competitively by Fe^{2+} , Co^{2+} and/or H_2PO_2^- to form CoFeP film. The increase of $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ facilitates the reduction of cobalt ion and H_2PO_2^- ion, which leads to an increase of Co and P content in the film. Also, the iron content in the film increase with the increase of FeSO_4 concentration.

Fig. 2 displays the XRD patterns of the samples. Careful analysis of these diffraction data shows that the crystalline nature of the films depends obviously on the bath composition and the post-treatment of the films. As shown in Fig. 2(a), a dispersion broad peak appears at approximately 44° for the film prepared with metallic salt ratio $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}/(\text{CoSO}_4 \cdot 7\text{H}_2\text{O}+\text{FeSO}_4 \cdot 7\text{H}_2\text{O})$ equal to 0, indicating typical amorphous structure of the CoP film. The half-widths of peaks decrease with the increasing metallic salt ratio, indicating the microstructure change of the films. The as-deposit films were exposed to heat treatment in vacuum at 200 and 400 °C, respectively. The XRD patterns of the as-deposit and the films after heat treatment are displayed in Fig. 2(b). From

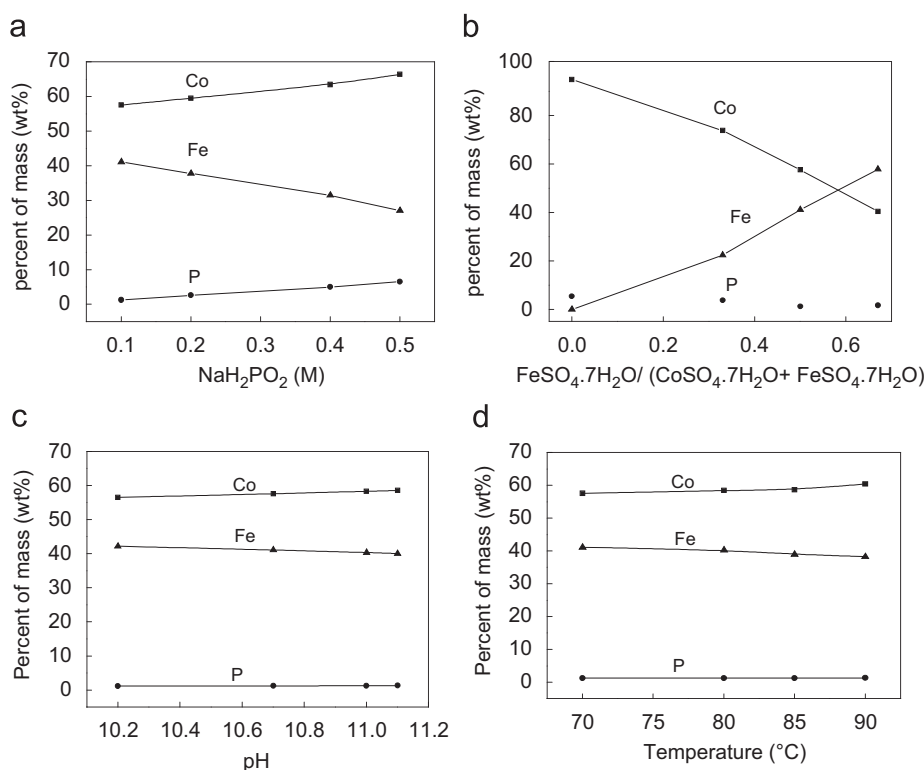


Fig. 1. Dependence of film composition on the bath composition and plating conditions.

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