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Enhanced performance of nanostructured FePtP alloy films for micro electro mechanical system applications

T. M. SELVAKUMARI[a,*], P. MUTHUKUMAR[b], S. GANESAN[c]
[a,b]*Department of Physics, Angel College of Engineering & Technology, Tirupur-641665, Tamilnadu, India*
[c]*Department of Physics, Government College of Technology, Coimbatore-641001, Tamilnadu, India*

In the present work, magnetic FePtP films were electrodeposited in the presence of urea in various concentrations. Effect of concentration of urea was studied. Vibration sample magnetometric studies indicate that urea has favourable impact on the magnetic properties of these films. Elemental composition of the molecules are studied using energy dispersive X-ray spectroscopy. The phosphorous content was found to be less than 10%. Morphology and structural properties are carried out using scanning electron microscopy and X-ray diffractometry. Reasons for variation in magnetic properties and structural characteristics are discussed. Mechanical properties such as residual stress, hardness and adhesion of the films are also studied. Our results indicate that nanostructured FePtP magnetic films is a promising candidate for micro electro mechanical system (MEMS) applications.

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1. Introduction

Magnetic micro-electro-mechanical-systems(MEMS) present a new class of conventional MEMS devices with great potential for science and applications. Using the same technology as for MEMS and incorporating magnetic materials as the sensing or active element offer new capabilities and open new markets within the information technology, automotive, biomedical, space and instrumentation. Magnetic MEMS are based on electromagnetic interactions between magnetic materials and active (coils) or passive magnetic field sources - permanent magnets [1]. Moreover, they are less susceptible to malfunction when subjected to adverse environments such as dust and humidity [2].

The face-centered tetragonal FePt phase, known as the $L1_0$ phase, is of interest for permanent magnet applications due to its excellent intrinsic magnetic properties [3]. Numerous studies have been carried out to develop hard magnetic films of this material due to potential application in high-density recording media and micro-electromechanical systems [4]. The elecrodeposited FePt samples showed a much smoother hysteresis loop than CoPt. The reason for this behaviour is due to the fact that the composition ratio may vary within a small range throughout the nanowires during electrodeposition [5]. The important magnetic properties of hard magnetic materials are remanent, coercivity and magnetic saturation. Electodeposited hard magnetic materials consist of heterogeneous alloys. Generally, hard magnetic alloys are iron based because fct-structured iron-platinum has a high magneto crystalline anisotropy [6]. As the formation of the L10 phase is kinetically hindered at room temperature, post annealing of the films is necessary. Electrodeposited and post annealed FePt and CoPt films can reach coercivities exceeding 1T [7]. The three phases present in FePtP alloy have a beneficial effect on the magnetic properties, i.e., FePt for the anisotropy, PtP_2 for pinning the magnetic domain wall, and the Fe-rich Fe-P phase for enhancing the magnetization, and their optimal combination can be a useful design tool for high performance magnets [8].

In the present study we investigated in detail the effects of concentration of urea and phosphorous source material on the magnetic, structural and mechanical properties of FePtP films.

2. Experimental details

FePtP films were electrodeposited on polycrystalline Cu substrate from a single bath containing : H_2PtCl_6 : 0.02M, $(NH_4)_2SO_4$: 0.1 M , $FeSO_4$:0.2M. Hereafter the above bath composition will be referred to as bath A. Then 0.2M and 0.4M of phosphorous acid (H_3PO_3) and 2.5 and 5.0 gl^{-1} of organic additive like urea were added in this bath and their effect on the properties of FePtP films were investigated. The solution pH was adjusted to 3 by adding a small amount of either sulfuric acid or hydrochloric acid. Films are deposited using dc plating in the current densities varying from 2-6 mA cm^{-2} at 60 minutes

The thickness of the deposits was tested using digital micrometer (Mitutoyo, Japan). Magnetic properties of deposited films were studied using vibrating sample magnetometry. X ray diffractometry (XRD) and scanning electron microscopy (SEM) were used to study the structure and morphology of these magnetic films respectively. From XRD data crystallite size of the deposited FePtP and film stress were calculated. Percentage of elements such as Fe, Pt and P present in the deposits was obtained using energy dispersive X-ray spectroscopy (EDS). Hardness of the deposit was obtained using Vicker's hardness tester using diamond intender method. Adhesion of the film was tested by bend and by scratch test. These tests are widely used in the field of electroplating [9].

3. Results and discussion

3.1 Magnetic properties

The effect of addition of urea into the bath-A along with H_3PO_3 was investigated using vibration sample magnetometer and the results are shown in Table 1. With the addition of low concentration of urea the deposit characteristics as well as its magnetic properties improved significantly. Under the best conditions involving addition of 0.2 M of H_3PO_3 and 2.5 g l^{-1} of urea at a current density of 6.0 mA cm^{-2} and time of deposition 60 minutes, the coercive and remanent values are 3000 Oe and 0.25 Am^2 respectively. The high coercivity obtained in FePt after annealing [10] is obtained in this case without annealing. With further increase in H_3PO_3 and urea concentration the coercive and remanent values are1000 Oe and 0.12 Am^2

Increase in magnetic properties of the films is mainly due to urea. The electrodeposited films were uniform and bright. The urea molecules thus are found to have leveling effect, which ensures uniform orientation of crystals during electrodeposition. On increasing the concentration of H_3PO_3 and urea magnetic properties of the films decreased. It was because of the stress present in the films, which was caused by the inclusion of decomposed products of additives.

Table 1. Effect of urea on the magnetic properties of FePtP films.

H_3PO_3 (M)	Urea (gl^{-1})	Magnetic saturation (Am^2)	Remanent (Am^2)	Coercivity (Oe)
0.2	0	0.8	0.16	1200
	2.5	0.64	0.25	3000
	5	0.69	0.21	1650
0.4	0	0.87	0.13	850
	2.5	0.72	0.17	1500
	5	0.79	0.12	1000

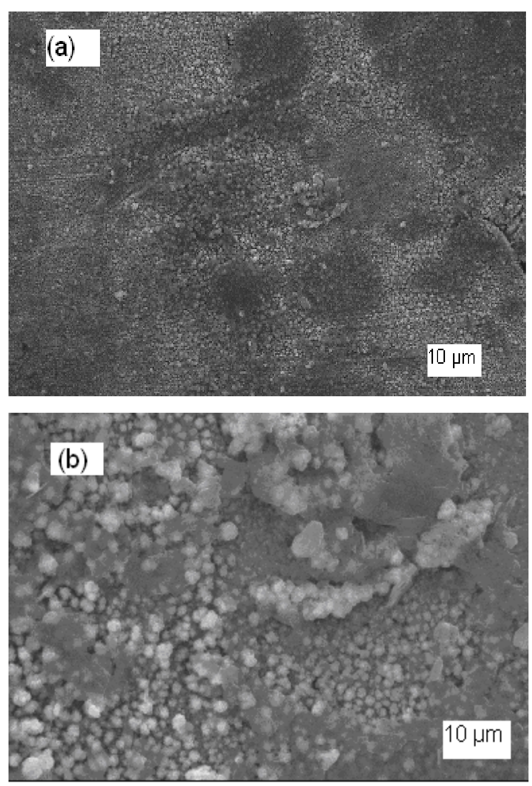
3.2 Surface analysis

Electrodeposited FePtP films were subjected to XRD studies. The X-ray wavelength used was 1.5405 A° of Cu Ka radiation. The result shows that FePtP films had face centered tetragonal structure and exhibited (111) plane primarily. (111) plane peaks were shifted because of the film stress. It was known that film stress will shift XRD peaks [11]. Stress of the films were calculated from XRD data using the formula i.e., Youngs modulus = stress / strain. The results are shown in the Table 2. Stress was low for film obtained from a bath contained 2.5 g l^{-1} of urea. It increased on increasing the concentration of urea to 5.0 g l^{-1}. This was due to the incorporation of decomposed products of additive in to the film. Crystallite sizes were also low for films obtained from 2.5 g l^{-1} urea. These were calculated from XRD data using the formula i.e., crystallite size = $0.9\ \lambda / \beta\cos\theta$ (Scherrer equation), where λ is the wavelength of X ray radiation, β is the peak full-width at half-height and θ is the diffracted angle. Crystallite sizes thus obtained were in the nano scale and it was shown in Table 2.

The micrographs of FePtP films subjected to SEM are presented in Fig. 1. In general microstructure of the FePtP was affected by the percentage of phosphorus content. Fig. 1 (b) and (e) shows the film with very low concentration of phosphorus, appeared to have a crevice pattern. The film obtained from a bath contained 5.0 g l^{-1} was cracked through substrate due to stress of the film as shown in Fig. 1 (c) and (f). It was also observed in Table 2 that film obtained from bath contained 5.0 g l^{-1} of urea had high stress.

Table 2. Effect of Urea on the structural and mechanical properties of FePtP film.

H_3PO_3 (M)	Urea (gl^{-1})	Crystalline Size(nm)	Stress (MPa)	Vickers Hardness (VHN)
0.2	0	35	148	380
	2.5	28	133	391
	5	31	140	360
0.4	0	39	157	358
	2.5	30	148	370
	5	34	153	350



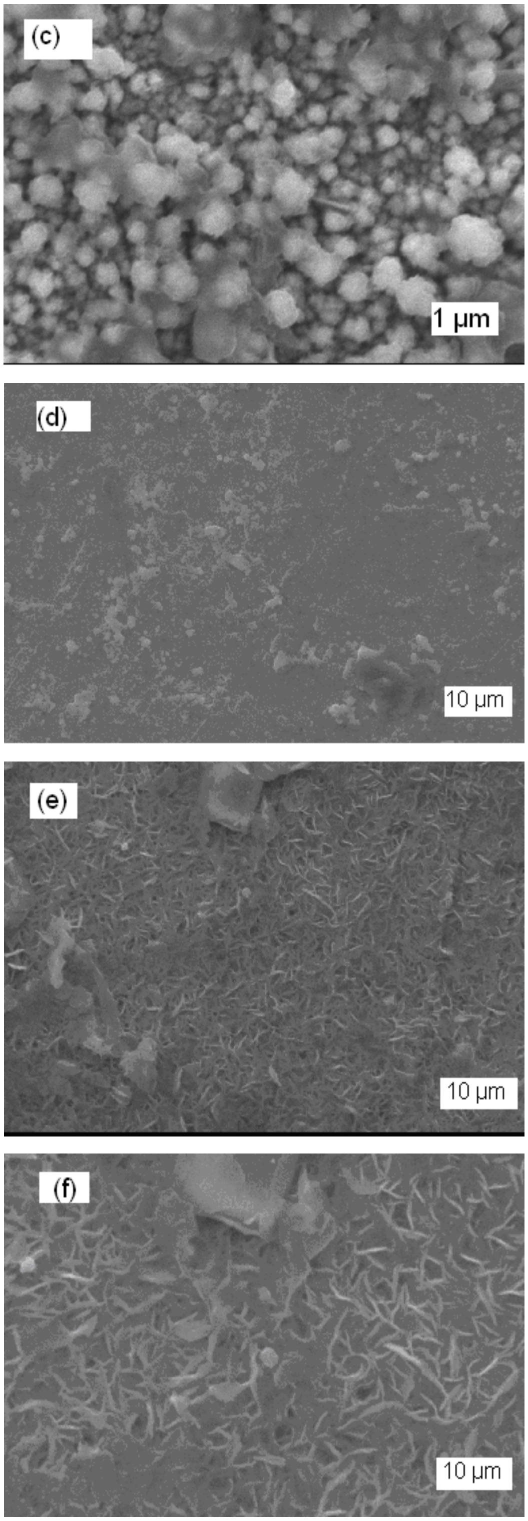


Fig. 1. SEM images of FePtP films from bath A with (a) H_3PO_3 : 0.2M, urea: 0 gl^{-1}, (b) H_3PO_3 : 0.2M, urea: 2.5 gl^{-1}, (c) H_3PO_3 : 0.2M, urea: 5.0 gl^{-1} (d) H_3PO_3: 0.4M, urea: 0 gl^{-1} , (e) H_3PO_3: 0.4M, urea: 2.5 gl^{-1}, (f) H_3PO_3 : 0.4M, urea: 5.0 gl^{-1}

3.3 Mechanical properties

FePtP films, which were selected for XRD and SEM studies, were tested for their Vicker's hardness number. The results are reported in Table 2. Higher concentration of urea in the bath decreased the hardness of the film. It was due to the stress present in the film, which caused cracks in the structure. Adhesion of the film with the substrate was found to be good.

3.4 Elemental analysis

Elements present in the film were analyzed by energy dispersive X-ray spectroscopy (EDX) and the results showed that the films with low phosphorous content have high magnetic properties. It was due to the addition of urea in the bath, which improved the crystalline structure of FePtP films.

4. Conclusions

A FePtP film with high hard magnetic properties can be obtained by galvanostatic electro deposition process. The high coercive value obtained in this work was 3000 Oe. Addition of urea increases the coercive value of the film. This is because the urea molecules are found to have leveling effect which ensures uniform orientation of crystals during electrodeposition. Increase in the concentration of phosphorous and urea will decrease the hard magnetic properties. It also increases the film stress which is a cause for cracked film. As these types of magnetic films are used in MEMS devices they should have minimum stress. 2.5gl^{-1} of urea was found to be the optimum concentration in the bath in order to obtain a FePtP film with improved magnetic, structural and mechanical properties.

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*Corresponding author. tms_kumari@rediffmail.com