Asian Journal of Science and Applied Technology
ISSN: 2249-0698 (P) Vol.1 No.1, 2012, pp.1-4
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DOI: https://doi.org/10.51983/ajsat-2012.1.1.691

Magnetic and Electroanalytical Properties of Electrodeposited FePtP Films

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(Received on 01 September 2011 and accepted on 02 March 2012)

Abstract - **FePtP films have been electrodeposited from a stable bath containing urea. By adjusting the current density and solution composition one can control the composition of the film. Effect of concentration of urea was studied. Vibration sample magnetometric studies indicate that urea has favourable impact on the magnetic properties of these films. Electroanalytical parameters like reduction potential and current for the FePtP deposition were studied using cyclic voltammeter. Reasons for variation in magnetic and electroanalytical properties are discussed.**

Keywords: **FePtP, Electrodeposition, Hard Magnetic Films, Magnetic Properties, Impedance**

I. Introduction

Magnetic micro-electro-mechanical-systems (Magnetic 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 microelectromechanical systems [4]. The electrodeposited FePt samples showed a much smoother hysteresis loop than CoPt. The reason for this behavior is due to the fact that the composition ratio may vary within a small range throughout the nanowires during electrodeposition [5,6]. The important magnetic properties of hard magnetic materials are remanent, coercivity and magnetic saturation. Electrodeposited 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 [7]. 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 [8]. 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 [9]. In the present study we investigated in detail the effects of concentration of urea and phosphorous source material on the magnetic and electroanalytical properties of FePtP films.

II. Experimental Details

A copper substrate of size 1.5 x 5.0 cm as cathode and pure steel of same size as anode were used for galvanostatic electrodeposition experiments. Current for electrodeposition was passed from a regulated direct current unit. Analytical reagent grade chemicals were used to prepare baths. An adhesive tape was used to mask off all the substrate except the area on which deposition of film was desired. Each substrate was buffed for removing scratches in a mechanical polishing wheel using a buffing cloth coated with aluminium oxide abrasive. Buffed substrates were degreased using acetone. Before electrodeposition these substrates were electrocleaned in an alkaline electrocleaning bath. The bath contained sodium hydroxide: 7.0 g/l; sodium carbonate: 20.0g/l; trisodium phosphate: 9.0g/l and sodium metasilicate: 24.0g/l. The bath was operated at 700 C and current density applied was 3.0 A/dm2. After electrocleaning the substrates were rinsed in distilled water. Electrodeposition was carried out on the cleaned substrates using different temperature, current density and time of deposition.

FePtP films were electrodeposited on polycrystalline Cu substrate from a single bath containing : H_2PtCl_6 : .02M, $(NH_4)_2SO_4$: 0.1 M , $FeSO_4$: .2M. Hereafter the above bath composition will be referred to as bath A. Then 0.2M and 0.4M of sodium hypophosphite (NaH_2PO_2) and 2.5 and 5.0 gl^{-1} of urea were added in this bath and their effect on the properties of FePtP films was 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.

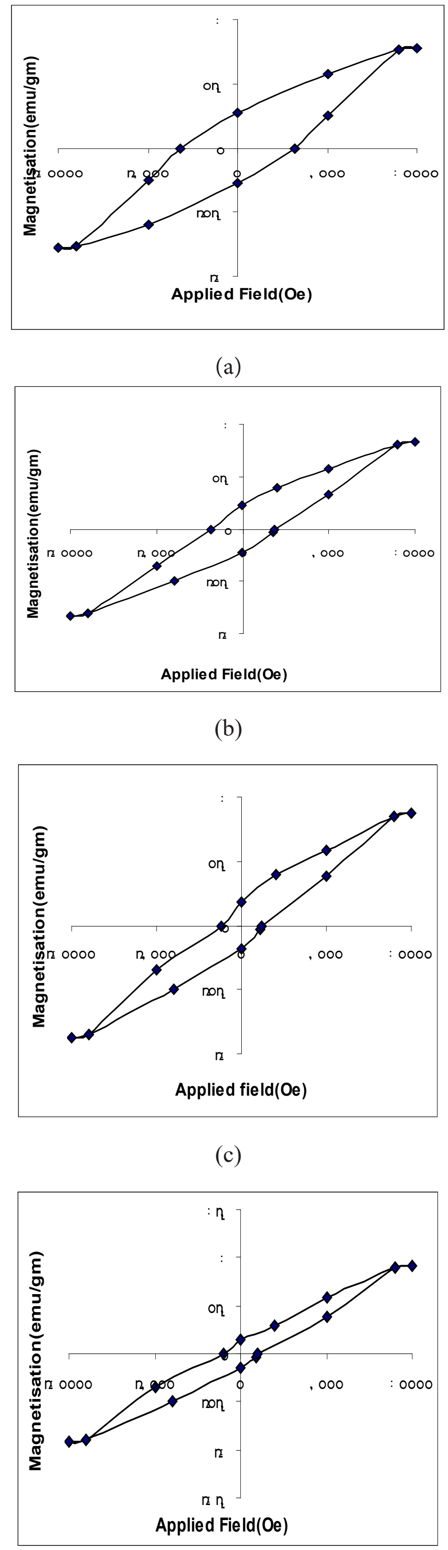
The thickness of the deposits was tested using digital micrometer (Mitutoyo, Japan). Magnetic properties of deposited films were studied using vibrating sample magnetometer. Cyclic voltammeter experiment was carried out for the optimized electrodeposition condition, for each organic additive using an electrochemical auto lab of model IM6 Germany.

III. Results And Discussion

A. Magnetic Properties and Thickness of the Deposit

FePtP deposits obtained from the galvanostatic electrodeposition studies were subjected to VSM in order to study the magnetic properties like coercivity, remanent and magnetic saturation etc. Hysteresis loops obtained from VSM are presented in Figure 1. These loops were obtained for FePtP films deposited from four different electrolytes with varying concentration of NaH_2PO_2 and urea at a current density of 6 mA cm^{-2} and for 60 minutes. From these M-H curves coercivity remanent, magnetic saturation, squareness was calculated. The results are presented in Table I. In all the concentration of NaH_2PO_2 and urea, the deposits obtained had higher coercivity than the deposits obtained from an electrolyte without organic additive. Similarly with increase in current density and time of deposition, the magnetic properties like coercivity, remanent and squareness were increasing predominantly and the magnetic saturation values decreased. This type of phenomena is exhibited by hard magnetic material. In general magnetic properties except saturation are directly proportional to the thickness of the deposits.

The deposits obtained from the bath having 0.4 M NaH_2PO_2 and 5.0 g l^{-1}urea had very low magnetic properties at high current density compared to deposits obtained from a bath having in 0.4 M NaH_2PO_2 only and not urea. This may be due to higher concentration of NaH_2PO_2. Even with the presence of urea the formation of $H_2PO_2^-$ ions was inevitable which causes reduction in thickness as well as magnetic properties. The decrease in magnetic properties is also because of higher concentration of urea. At higher concentration of urea the decomposed product of urea gets into the deposits and thereby making the deposits have more stress.



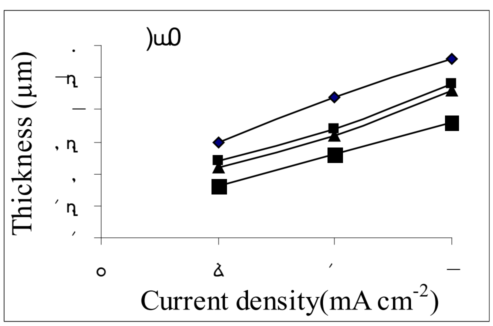
(a) (b) (c) (d)

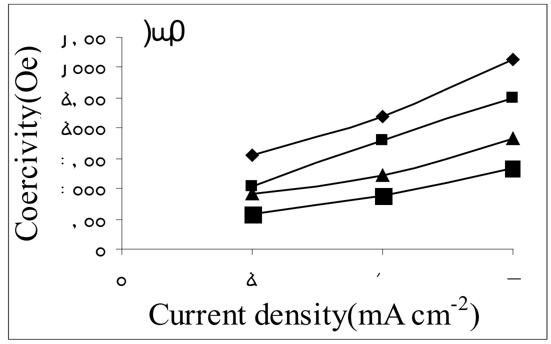
Fig.1 Hysteresis loops of FePtP film from Solution: A with (a) NaH_2PO_2 : 0.2 M ; Urea: 2.5 g l^{-1}

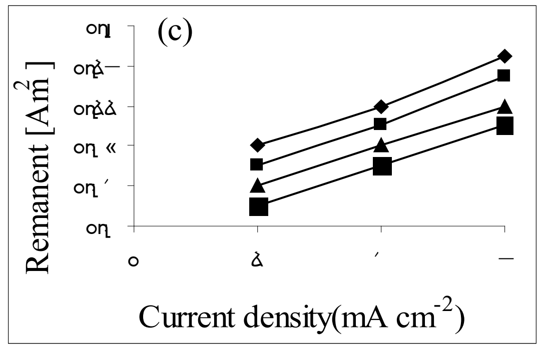
(b) NaH_2PO_2 : 0.2 M ; Urea: 5.0 g l^{-1} (c) NaH_2PO_2: 0.4 M ; Urea: 2.5 g l^{-1} (d) NaH_2PO_2 : 0.4 M ; Urea: 5.0 g l^{-1}

Variation of thickness and magnetic properties with respect to current density is presented in Figures 2 (a), (b), (c) and (d). Fig. 2 (a) gives thickness increases with increase in current density. Change in coercivity of the deposit with respect to current density shows that with increase in the concentration of NaH_2PO_2 the coercivity decreases as shown in 2 (b). The deposit obtained from bath having 0.2 M NaH_2PO_2 and 2.5 g l^{-1} of urea have a high coercivity. The deposit obtained from the bath having 0.4M NaH_2PO_2 and 5.0 g l^{-1} urea have very low coercivity values. However, in general the increase in current density increases coercivity.

Figure 2(c) presents the relation between remanent with current density and remanent. However when the electrodeposition was carried out for 60 minutes from a bath containing 2.5 g l^{-1} of urea the remanent values increased. This may be due to the fact that on increasing the time of deposition, the crystalline of the film distorted on the surface of the film. Effect of current density on the magnetic saturation of the films is presented in Figure 2(d). In contradiction to other magnetic properties, magnetic saturation decreases with increase in current density and. This can be indirectly correlated to the coercivity of the films ie on increasing the current density coercivity of the film increases. This substantiates the point that hard magnetic material having high coercivity will have low magnetic saturation.







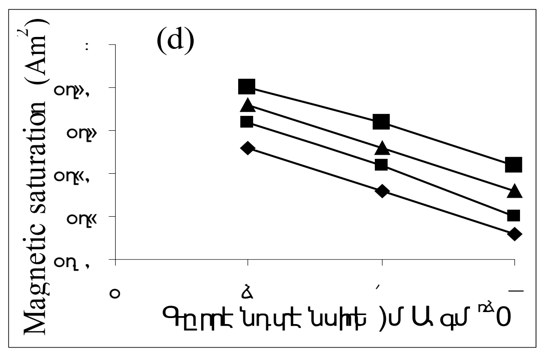


Fig 2. Effect of current density on thickness, coercivity, remanent and magnetic saturation of FePtP films electrodeposited from bath A with:
(▪) NaH_2PO_2 : 0.2M; Urea : 2.5 gl^{-1},
(■) NaH_2PO_2 : 0.2M ;Urea : 5.0 gl^{-1}, (▲) NaH_2PO_2 : 0.4M; Urea : 2.5 gl^{-1},
(■)NaH_2PO_2: 0.4M; Urea : 5.0 gl^{-1}

TABLE I THICKNESS AND MAGNETIC VALUES OF FEPTP FILMS ELECTRODEPOSITED FROM SOLUTION: A WITH VARYING CONCENTRATION OF NaH_2PO_2 AND UREA

Current density ($mAcm^{-2}$)	NaH_2PO_2 (M)	Urea (gl^{-1})	Thickness of deposit (µm)	Magnetic saturation (emu)	Remanent (emu)	Coercivity (Oe)	Squareness
2	0.2	2.5	5.5	.88	.18	1550	.19
		5	6.1	.91	.16	1050	.17
	0.4	2.5	5.2	.94	.13	900	.13
		5	5.6	.98	.11	650	.10
4	0.2	2.5	6.2	.83	.22	2200	.25
		5	6.7	.88	.17	1800	.22
	0.4	2.5	5.8	.92	.15	1220	.19
		5	6.3	.95	.12	880	.14
6	0.2	2.5	6.8	.78	.27	3150	.32
		5	7.3	.83	.23	2500	.26
	0.4	2.5	6.4	.87	.18	1820	.23
		5	6.8	.91	.14	1350	.18

B. Electro Analytical Properties

1. Cyclicvoltametric Studies

To study the electroanalytical parameters like reduction potential and current for the FePtP deposition cyclic voltametry was used. It was carried out from an electrolyte consists of solution:A, NaH_2PO_2 :0.2 M and Urea:2.5 g l^{-1} using a three electrode cell. Voltamogramme was recorded at 50 mVs^{-1} slew rate. A well defined reduction peak is observed at -650 mV s^{-1} vs. SCE. It is measured from the graph in Fig.3. In the reverse scan there is a broad peak is observed at +400 mV. The above peaks are representing electrodeposits and electro dissolution of the alloy. In the alloy electrolyte Fe, Pt and P codeposits simultaneously over whole of the negative potential region which indicates this electrodeposition is kinetically favoured.

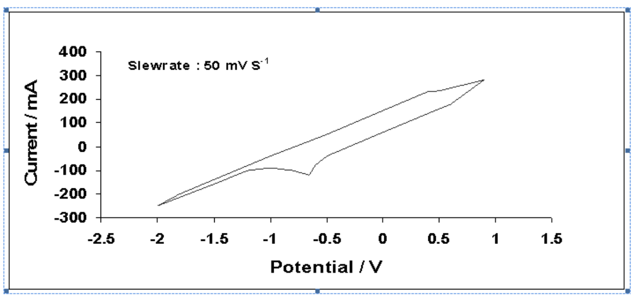


Fig. 3 Cyclic Voltammogramme graph of FePtP films electrodeposited from solution : A, NaH_2PO_2 : 0.2 M and urea 2.5 gl^{-1}

2. Impedance Properties

The major application of FePtP hard magnetic film is in MEMS devices. MEMS fabrication involves varies corrosive chemicals such as HF etc, for etching, the film incorporated in MEMS should be highly corrosion resistance.

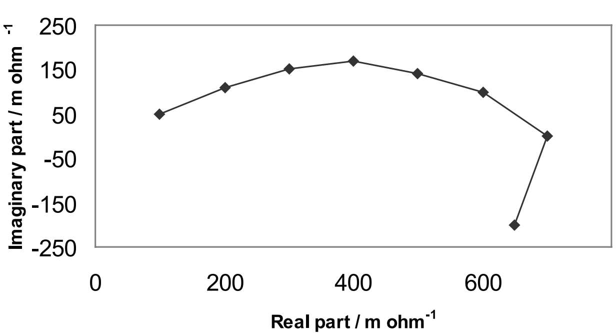


Fig.4 Impedance graph of FePtP films electrodeposited from solution : A with NaH_2PO_2 : 0.2 M and urea 2.5 gl^{-1}

In order to study the corrosion resistance of the deposit obtained in present study Nyquist plot was obtained for FePtP film. This film was electrodeposited at 6.0 mA cm^{-2} for 60 minutes from a bath solution:A, NaH_2PO_2:0.2M and urea 2.5 g l^{-1}. It was already understood that the magnetic properties of the film was high at 2.5 g l^{-1} of urea. The charge transfer resistance (R_{ct}) value obtained from the Nyquist plot shows in Fig 4 is 600 ohm cm^2.

IV. Conclusion

Galvanostatic electrodeposition of FePtP films with superior magnetic properties like coercivity in the order of 3150 Oe, was obtained in this experiment. High concentration of urea and NaH_2PO_2 is not desirable because of the decrease in the magnetic properties. This is mainly because of the high stress present in the film. At higher concentration of urea some of the decomposed products also get accumulated in the deposit structure leading to an increase in the stress of the deposit. Cyclic voltametric results revealed that these deposition is kinetically favored. In the presence of lower concentration of NaH_2PO_2 and urea electrodeposition was carried out smoothly and it was observed in cyclic voltammetry also as well defined electrodeposition peak at -650 vs SCE. Nyquist plots for the FePtP deposits obtained from the bath with low NaH_2PO_2 and urea showed high corrosion resistance.

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