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Magnetic Properties and Microstructural Characterisation of Nanostructured FePt Films Fabricated by Electrodeposition

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Abstract

The study of magnetic and microstructural characteristics of some nanostructured magnetic thin layers have fundamental importance to understand magnetization dynamics and is related to applications in micro electro mechanical system applications. This paper is devoted to the investigation of FePt alloy films by electrodeposition technique. High quality of deposit with low cost is the advantage of this technique. Structural analysis of the film was carried out using X-ray diffractometry & scanning electron microscopy. Elemental compositions of the film were studied using energy dispersive X-ray spectroscopy. Magnetic properties were studied using vibration sample magnetometer. The surface morphology, crystalline structure, grain size and magnetic properties of the plated films have been compared. The X ray analysis and magnetic measurements reveal the presence of hard and soft magnetic phase with various temperature for dc plated films. Thickness was measured by using digital micrometer. Hardness and adhesion of the film were also studied. Testing results shows that magnetic and structural properties depends upon the bath temperature, current density, annealing and composition ratio of Fe and Pt. Films are shiny and smooth. Reasons for variation in magnetic properties and structural characteristics were discussed.

Keywords: Fept, Electrodeposition, Hard mangetic film, Magnetic properties.

Introduction

With the progress in the field of Micro Electro Mechanical System (MEMS) technologies[1-5] there has been growing interest in developing electroplated, nanostructured soft and hard magnetic materials[6,7] for microactuators, micromotors and microswitches. The possibilities of these electroplated materials, retaining hard and soft magnetic properties up to several microns thickness, gives researches opportunity to explore them for micro fabrication of MEMS devices. Recently, much effort is being made to electrodeposits also materials of the group of L10 ordered alloys, like FePt[8,9] and CoPt[10,11], because they exhibit a significantly higher uniaxial magneto crystalline anisotropy. 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[11].

In the present study we investigated in detail the effects of bath temperature, current density and composition ratio of electrodeposited magnetic FePt films. Also we discussed their structural and magnetic characterization.

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 sodum 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 70^0 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.

FePt films were electrodeposited on polycrystalline Cu substrate from a single bath containing 1 mmol/ l H_2PtCl_6, 0.1 mol/l $(NH_4)_2$ SO_4 and 0.3 mol/l $FeSO_4$. The solution pH was adjusted to 3 by adding a small amount of either sulfuric acid or hydrochloric acid.

The thickness of the deposits was tested using digital micrometer (Mitutoyo, Japan). Magnetic properties of deposited films were studied using vibrating sample magnetometry. In this technique the material under study was contained in a sample holder, which was centered in the region between the pole pieces of a laboratory magnet. A slender vertical sample holder, which was centered in the region between the pole pieces of a laboratory magnet. A slender vertical sample rod connects the sample holder with a transducer assembly. The transducer converts a sinusoidal alternating current drive signal into a sinusoidal vertical vibration of the sample rod.

Coils mounted on the pole pieces of the magnet pick up the signal resulting from the sample motion. 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 FePt and film stress were calculated. Percentage of elements such as Fe and Pt present in the deposits were obtained as follows. For elemental analysis FePt film was electrodeposited on stainless steel substrate to ensure easier peeling off of the film. After deposition the film was peeled off from the substrate. It was dissolved in 3:1 v/v of H_2SO_4 and HNO_3 and the percentage composition 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 test (bending the film with substrate to 180^0) and by scratch test (draw equal lines by pin and paste an adhesive tape over the scratch and pull it. If the film comes with tape then adhesion is poor).

Results and discussion

Surface analysis

Morphological observation

Electrodeposited FePt films obtained from the bath temperatures 30^0C, 50^0C, 70^0C, 90^0C and after annealing at 155^0C in vacuum for 30 minutes were subjected to SEM. The micrographs are presented in Fig 1. The results shows that the film deposited at low temperature appeared crevice pattern with crack, grains are formed in random order and it also appears less bright. In the film deposited at high temperature as well as after annealing grain sizes are visible & very clear in granular form. They are in more order with fct structure.

Structural analysis

Electrodeposited FePt films were subjected to XRD studies. The X-ray wavelength used was 1.5406 A^0 of Cu Kα radiation. Films obtained from various bath temperatures like 30^0C, 50^0C , 70^0C, 90^0C and after annealing at 155^0C in vacuum for 30 minutes were studied for their structural characteristics as shown in Fig 2. FePt films had face centered tetragonal structure and exhibited (111) plane predominantly. Stress of the films were calculated from XRD Pattern using the formula: Youngs modulas= stress/strain and presented in Table 1. After annealing stress value decreases. It is shown in in Table 2.

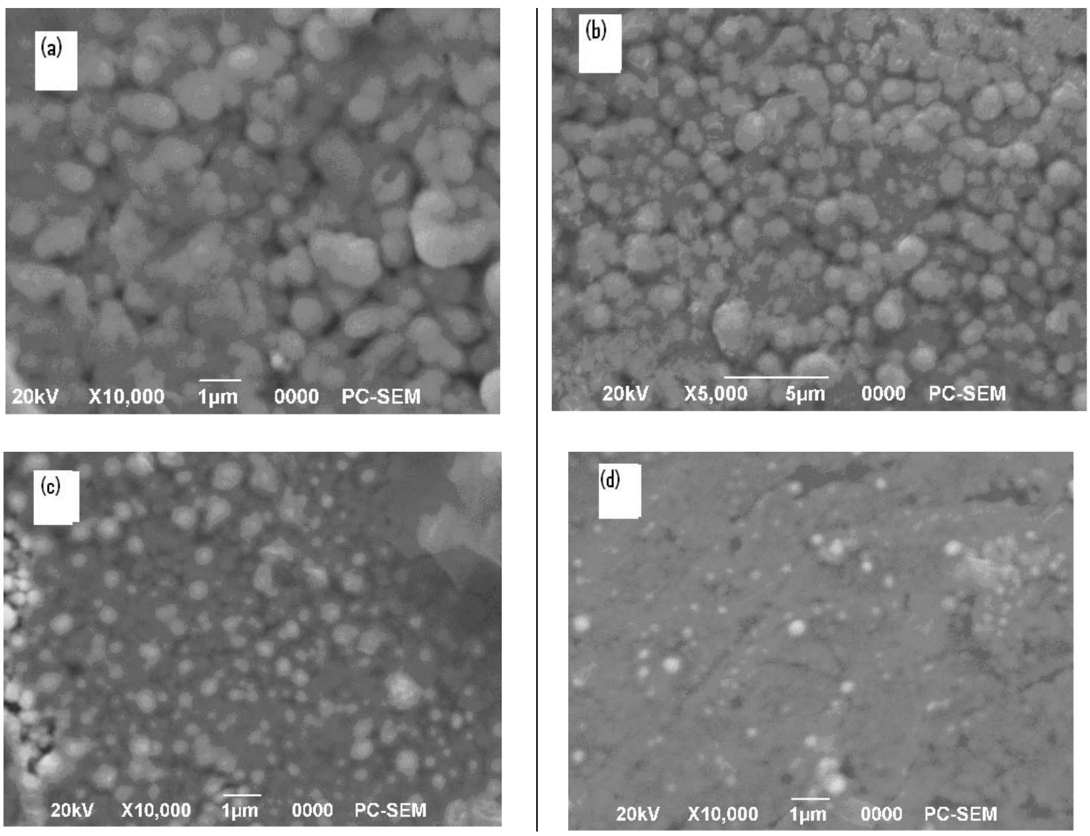
Crystalline sizes were calculated from XRD data using the formula: crystalline size=0.9λ / $\beta\cos\theta$ .The results obtained were in the nano scale and it was shown in Table 1. After annealing at 155^0c in vacuum for 30 minutes the crystalline size is decreased and is shown in Table 2.

Table 1: Structural and Mechanical properties of FePt films electrodeposited at various bath temperatures for 30 min in the current density of 5 mA/cm^2.

Temperature ^{0}c	Crystalline size(nm)	Stress(MPa)	Vickners Hardness(VHN)	Film composition (wt%) Fe Pt
30	39	187	259	15 85
50	37	178	268	18.4 81.6
70	34	155	298	20 80
90	28	133	330	29.5 70.5

Table 2: Structural and Mechanical properties of electrodeposited FePt films after annealing at 155^{0}c .for 30 min in vacuum in the current density of 5 mA/cm^2.

Temperature ^{0}c	Stress(MPa)	Crystalline size(nm)	Vickers Hardness(VHN)
50	150	33.5	300
70	136	29	322
90	118	22	346



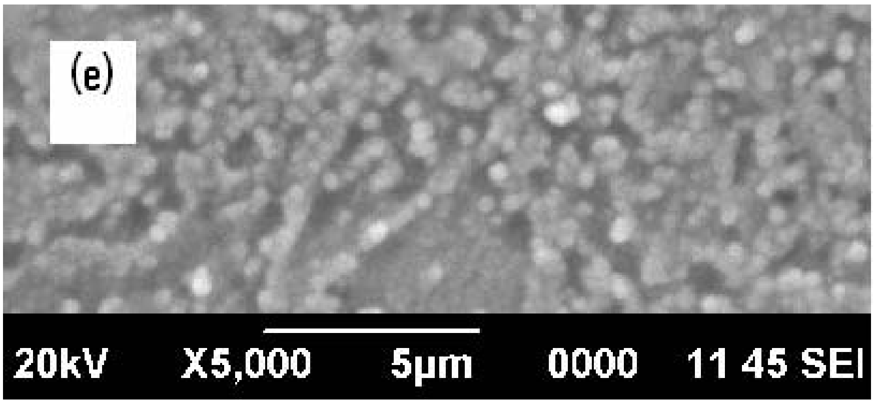
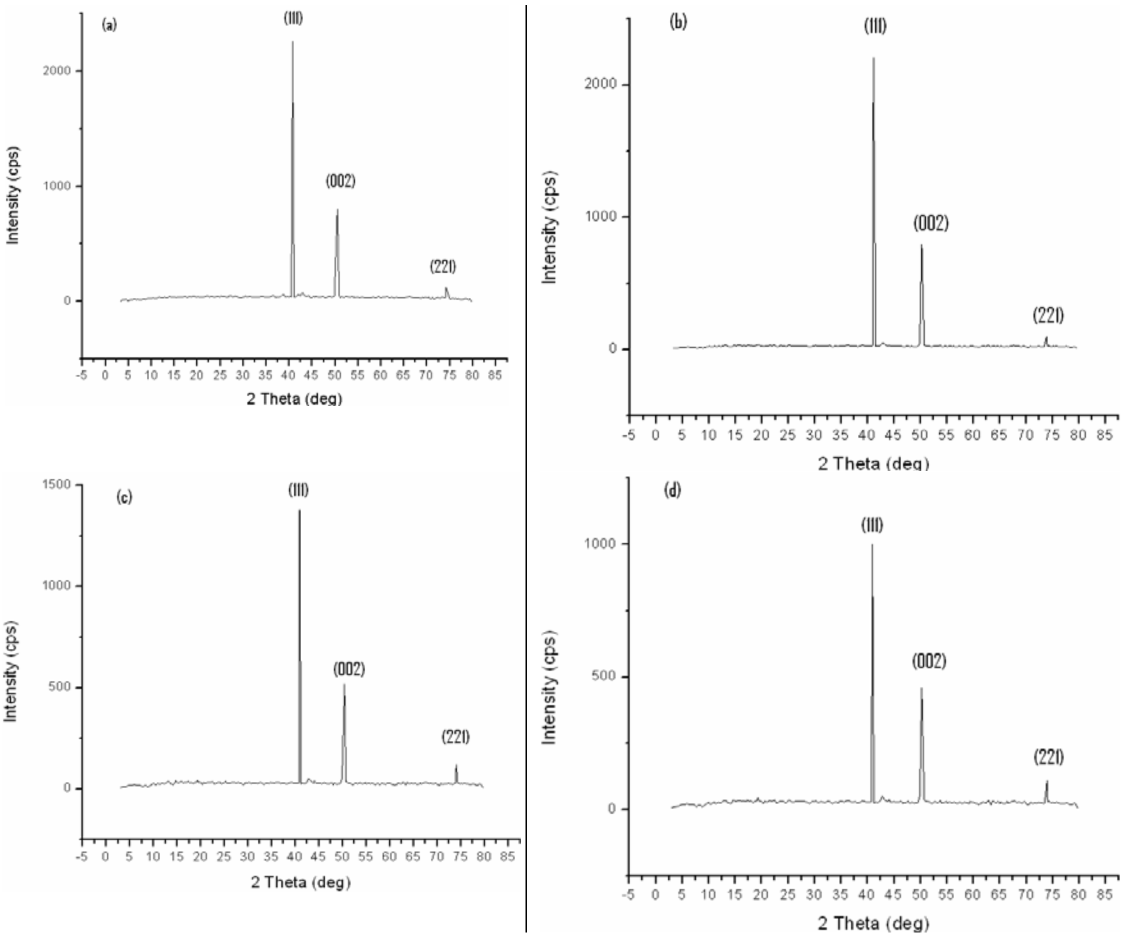


Figure 1: SEM images of FePt films electrodeposited for 30 min the current density of 5 mA/cm^2 at temperatures (a)30^{0}c, (b)50^{0}c, (c)70^{0}c, (d) 90^{0}c, (e) after annealing sample (b) at 155 ^{0}c for 30 min in vacuum.



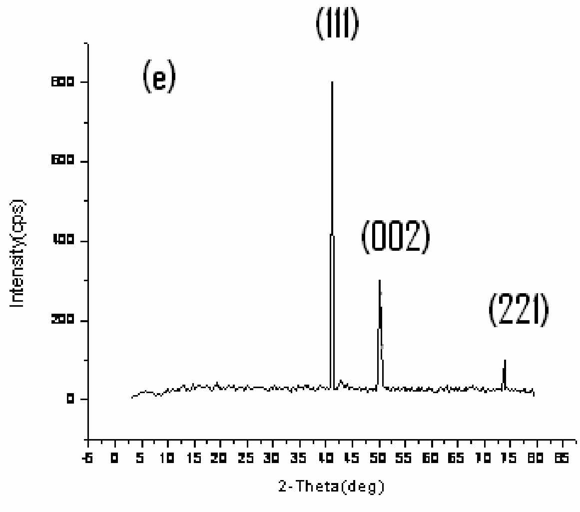


Figure 2: XRD patterns for electrodeposited FePt film at current density:5mA/cm^2 Deposition time: 30 min in Temperatures: (a)30^{0}c, (b) 50 ^{0}c, (c)70^{0}c, (d) 90^{0}c and (e) after annealing sample (b) at 155^{0}c in vacuum for 30 min.

Mechanical properties

Adhesion of the film with the substrate is tested by bend test and scratch test. It showed that the film is having good adhesion with the substrate. Hardness of these films was examined using a Vicker's hardness tester by the diamond intender method. The results are reported in Table 1. The results shows that hardness increases with increasing bath temperature.

Elemental analysis

Elements present in the film were analysed by energy dispersive X-ray spectroscopy (EDS) and the results are presented in Table 1. It showed that the films obtained from high bath temperatures have low phosphorus content and their magnetic properties are high.

Thickness and magnetic properties

Magnetic properties of electrodeposited films were studied using Vibration Sample Magnetometer. Magnetic properties are strongly dependent on bath temperature , film thickness and annealing. The coercivity of electrodeposited FePt films increased with increase in bath temperature and annealing. Table 3 summarizes the effect of bath temperature and current density on the thickness & magnetic properties of electrodeposited film before annealing. Thickness of the deposit slightly increases with increase in bath temperature and also with the increase in current density. Magnetic saturation decreases with increase in temperature. But coercivity and remanent increases with increase in temperature. Table 4 presents the effect of bath temperature and current density on the magnetic properties of electrodeposited film after annealing. This result shows that coercivity increases after annealing at 155^{0}C for 30 minutes in vacuum i.e the as deposited film is a soft magnet and becomes a hard magnet at high bath temperature and after annealing.

As the average crystallite size of these films are in the nano scale, considerable changes in the magnetic behaviour can occur. When the crystallite size is reduced to the extent that the domain wall thickness is comparable to the crystallite size, the coercivity is found to decrease. But in the present work the coercivity increases when crystallite size approaches high nano level. Analysis of crystalline size, microstructure, and magnetic properties confirm that the origin of magnetic properties is because of the strongly interacting array of single domain crystals. This is mainly due to the films at high bath temperature and annealing.

Table 3: Effect of bath temperature and current density on the thickness & magnetic properties of electrodeposited film before annealing at the deposition time of 30 min in the current density 5 mA/cm^2.

Current Density mA/cm^2	Temperature ^{0}c	Thickness of deposit µm	Magnetic saturation (emu)	Remanent (emu)	Coercivity Oe	Squareness
2.5	30 50 70 90	2.3 2.4 2.5 2.6	.77 .72 .69 .65	.03 .04 .07 .10	300 450 700 900	.07 .09 .10 .12
5	30 50 70 90	2.8 2.9 3.1 3.4	.72 .67 .61 .55	.09 .11 .13 .16	600 760 950 1100	.10 .13 .15 .17
7.5	30 50 70 90	3.4 3.5 3.6 3.7	.68 .64 .59 .52	.13 .15 .18 .21	1100 1300 1500 1900	.14 .16 .17 .19

Table 4: Effect of bath temperature and current density on the magnetic properties of electrodeposited film after annealing at 155^{0}c .for 30 min in vacuum at the deposition time of 30 min in the current density of 5 mA/cm^2.

Temperature ^{0}c	Thickness of deposit	Magnetic saturation Am^2	Remanent Am^2	Coercivity Oe	Squareness
50	2.9	.54	.17	1590	.20
70	3.1	.51	.19	1700	.23
90	3.4	.49	.21	1950	.25

Conclusion

We have presented the report of continuous electrodeposited FePt film. The bath required for electrodeposition contained 1mmol/ l H_2PtCl_6, 0.1 mol/l $(NH_4)_2$ SO_4 and 0.3 mol/l$FeSO_4$. The $L1_0$ phase was developed at high bath temperature and after annealing at 155^0c i.e as deposited film was a soft magnet and becomes a hard magnet after annealing. The film deposited at low temperature appeared crevice pattern with crack, grains are formed in random order and it also appears less bright. In the film deposited at high temperature and after annealing the grain sizes are visible & very clear in granular form i.e the microstructural studies showed that the ordered fct phase is formed at high bath temperature and after annealing. This method using a novel bath when compared to vapor deposition methods, opens an alternative route for the production of FePt films that may be useful for MEMS applications.

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