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Synthesis and characterization of ferrous manganese tungsten thin films for magnetic MEMS devices

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Fe-Mn-W thin films were synthesized by electrodeposition technique with different current density and different deposition time. Electrodeposition method is a flexible and less expensive avenue which produces alloys in controlled thickness, shapes and sizes. The hysteresis loops of Fe-Mn-W alloys films were studied by using vibrating sample magnetometer. The crystalline size and surface morphology of the deposited thin films were calculated by using X-ray diffraction (XRD) studies and Scanning Electron Microscope (SEM). Energy Dispersive X-ray Spectroscopy (EDAX) was used to identify the components of the films. Hardness and adhesion of the deposited thin films were investigated by Vickers hardness tester using diamond intender method.

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1. Introduction

Electrodeposition method has become a presiding manufacturing technology because of its low-cost, good composition control, simple scale-up, significant flexibility with substrate compatibility, easy to maintenance, high quality deposits and also high quality thin films [1, 2]. Ferro magnetic materials like Fe, Ni and Co play superior role in micro electromechanical system (MEMS) devices, nano electromechanical system (NEMS) devices including actuators, magnetic sensors, magnetic recording head, transformers and data storage [3-7]. Compared to Nickel and Cobalt, Ferrous, Manganese, Tungsten coating is substantially cheaper and also characterized by higher physical-chemical properties compared to pure iron [8]. Iron-tungsten alloys have higher wear resistance than pure iron. Addition of tungsten into metals withdrawing of deposited films provide good properties of coatings, high mechanical strength, apparently increased the high corrosion resistance, heat resistance and unique properties that can be utilized in the electronic, automobile industries, rocketry and space technology [9-12]. The Ferrous Sulphate bath create a film with good adhesion to the substrate that is smooth and light grey in colour.

In the current study, the effects of current density on the magnetic properties of ferrous manganese tungsten films were studied. The crystal structure, morphology, magnetic properties, hardness and EDAX analysis of Fe-Mn-W films were explored.

2. Materials and experimental procedures

Fe-Mn-W thin film was effectively coated by using electrodeposition method. 1.5 x 5 cm size of copper substrate was used as cathode and the same size of stainless steel plate was used as anode for galvanostatic electrodeposition method. By using regulated power supply, the films were

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deposited. The copper substrate and stainless steel plate were cleaned by using concentrated sulphuric acid (H_2SO_4) and were rinsed with deionnized water to remove the impurity particles on the substrate. After that they were again cleaned with acetone to remove the impurity particles completely from electrodes and finally it was completely rinsed with doubly distilled water.

Electrodeposition of Fe-Mn-W magnetic thin film was coated from, Ferrous sulfate ($FeSO_4.7H_2O$) 0.1 M, Sodium tungstate ($Na_2WO_4.2H_2O$) 0.05 M, Manganese II Sulphate ($MnSO_4.H_2O$) 0.1 M, Trisodium citrate ($Na_3C_6H_5O_7.2H_2O$) 0.3 M, Ammonium sulphate ($(NH_4)_2SO_4$) 0.3 M, Boric acid (H_3BO_3) 0.16 M. The pH value of electrodeposition bath was maintained at 8. The properties of Fe-Mn-W films were investigated with different current densities (20 mA cm^{-2}, 25 mA cm^{-2} and 30 mA cm^{-2}) and various deposition time (15, 30, 45 minutes)

2.2 Characterization

Digital micrometer (Mitutoyo, Japan) was used to measure the thickness of the films. X-ray diffractometer (Rich Seifert, model 3000) and scanning electron microscope (JEOL) were used to analyze the structure and morphology of the magnetic films. The existence of elements like Fe, Mn and W were obtained by energy dispersive X-ray analysis (EDAX). The crystalline size, strain and dislocation density were determined by X-ray diffractometry (XRD). Vickers hardness tester through diamond intender method was used to find the hardness of the deposited thin film. Bend test and scratch test were used to test the adhesion of the films. Vibrating sample magnetometer (VSM) was used to study the magnetic properties such as magnetic saturation (M_s), coercivity (H_c) and retentivity (M_r).

3. Results and discussion

3.1 Thickness Study

Table 1 shows the effect of current density and deposition time on the thickness and the magnetic properties of Fe-Mn-W films by electrodeposition method. The thicknesses of Fe-Mn-W films were increased with increase of current density and deposition time. The thickness of the films were increased from 2.6 µm to 3.9 µm at deposition time of 45 minutes when the current density was increased from 20 mA cm^{-2} to 30 mA cm^{-2}.When current density and deposition time increased, the coercivity and remanent polarization of the magnetic film also increased.

Table 1: Effect of the thickness and magnetic properties of Fe-Mn-W thin films electrodeposited for different deposition time and current densities.

S. No.	Current density (mA cm^{-2})	Deposition Time (mins)	Thickness of deposit (µm)	Magnetic Saturation (x 10^{-3}emu)	Remanent polarization (x 10^{-3}emu)	Coercivity (Oe)	Squareness
1		15	2.6	16.608	2.032	96.426	0.122
2	20	30	2.8	09.978	0.357	99.726	0.036
3		45	3.3	19.333	2.050	111.47	0.106
4		15	3.1	19.383	2.694	102.07	0.139
5	25	30	3.3	7.222	0.560	123.50	0.078
6		45	3.4	32.429	6.258	121.61	0.193
7		15	3.5	20.092	2.952	121.18	0.147
8	30	30	3.8	10.67	1.867	140.38	0.175
9		45	3.9	33.981	9.316	147.67	0.274

3.2 Surface analysis

3.2.1 Structural analysis

Figure 1 shows an XRD analysis of Fe-Mn-W films that were deposited at various current densities. The observed XRD patterns of Fe-Mn-W films were compared with the standard JCPDS data and were found to have Hexagonal structure in Fe_2W (203) plane (JCPDS card No:03-0920), cubic structure with Fe (222) plane (JCPDS card No:01-1267) and Hexagonal structure in Fe (008) plane (JCPDS card No:50-1275). (203) predominant peak was the strongest one when compared with the other observed peaks. The sharp peak present in XRD patterns exhibited good crystalline structure. Average crystalline size (D) of Fe-Mn-W films were calculated by using Scherrer equation.

$$\text{Crystalline size (D)} = k\lambda/\beta\cos\Theta \tag{1}$$

Where, D is the size of the crystalline, λ is the wavelength of the incident X-ray beam used for measurements and β is the full width half maximum. Crystalline size of the film decreased with increase of current density [9]. The dislocation density (δ), micro strain (ε) were obtained by equations (2&3) and the results were registered in Table 2.

$$\text{Dislocation density}(\delta) = 1/D^2 \tag{2}$$

$$\text{Micro strain}(\epsilon) = \beta\cos\Theta/4 \tag{3}$$

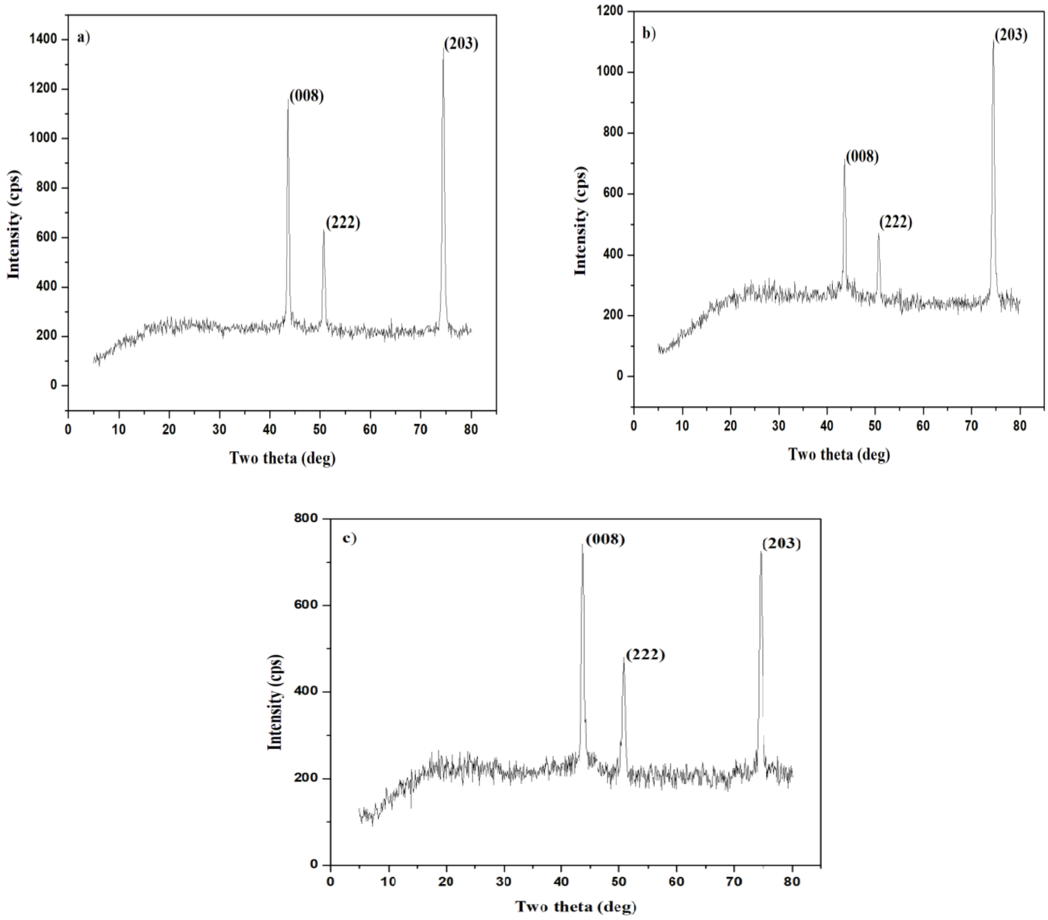

Fig. 1. XRD images of Fe-Mn-W films electrodeposited for 45 minutes at (a) 20 mA cm^{-2} (b) 25 mA cm^{-2} (c) 30 mA cm^{-2}

Table 2. Crystalline size, hardness and composition of Fe-Mn-W films for different current densities at the deposition time of 45 minutes.

Current density (mA cm^{-2})	Crystalline size (nm)	Strain 10^{-4}	Dislocation Density ($10^{14}/m^2$)	Vicker Hardness Number (VHN)	Film Composition (at %)		
					Fe	Mn	W
20	18.020	4.293	3.098	161.45	74.28	0.54	25.18
25	17.577	4.408	3.240	169.18	75.97	0.63	23.40
30	16.634	4.685	3.648	178.22	76.67	0.30	23.03

Table 2 shows that dislocation density (δ) and Micro strain(ϵ) were increased with increasing of current density. The Crystalline sizes of the deposited films were obtained in the nano scale range and the results were shown in Table 2.

3.2.2 Surface morphology analysis

Figure 2 shows SEM images of Fe-Mn-W deposited films with different current densities 20 mA cm^{-2}, 25 mA cm^{-2}, 30 mA cm^{-2} at 45 minutes. The crystalline of Fe-Mn-W magnetic film predominantly depend on the amount of ferrous, manganese and tungsten. SEM image clearly shows the gray color film with smooth and homogeneous surface at low current density such as 20 mA cm^{-2}, 25 mA cm^{-2}, micro cracks were formed at 30 mA cm^{-2}due to the presence of tungsten alloy and internal stresses were built up. The results are shown in the Figure 2(a), 2(b) and 2(c) respectively.

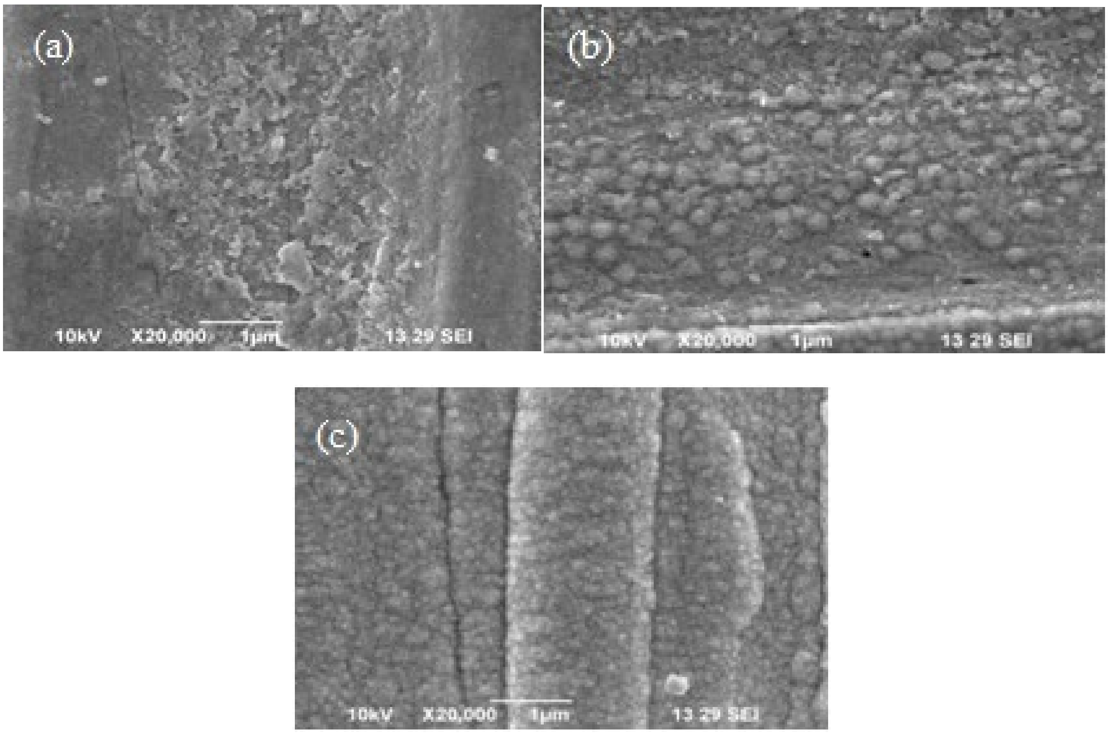

Fig. 2. SEM images of Fe-Mn-W films electrodeposited for 45 mins at a) 20 mA cm^{-2} b) 25 mA cm^{-2} c) 30 mA cm^{-2}

3.3 Mechanical properties

Hardness of Fe-Mn-W electrodeposited thin films were measured by using Vickers hardness tester. Table 2 showed the hardness values of magnetic films at current densities 20 mA cm^{-2}, 25 mA $cm^{-2}$and 30 mA cm^{-2}at 45 minutes of deposition time. From Table 2**,** hardness of the electrodeposited film mainly depends on current density. When current density was increased, hardness of the films also increased [9]. Adhesion of the Fe-Mn-W films on the Cu substrate were tested by using scratch test and bend test. The test exposed that Fe-Mn-W films have been adhered well on the Cu substrate.

3.4 Elemental composition analysis

Composition of electrodeposited Fe-Mn-W thin films were analyzed by Energy Dispersive X-Ray Analysis. The EDAX data of Fe-Mn-W thin films are shown in Table 2. From the study of EDAX, it was concluded that the films which have above 74% of Fe element have high magnetic property. The percentage of ferrous increased at the same time the percentage of tungsten decreased with increase of current densities [4].

3.5 Magnetic properties

The magnetic properties of the Fe-Mn-W electrodeposited films were obtained from vibrating sample magnetometer. Squareness of the films were calculated by using the formula,

$$\text{Squareness} = \text{Retentivity}/\ \text{Magnetic saturation}$$

The value of magnetic saturation (emu), coercivity (Oe) and squareness values were tabulated in Table 1. VSM images of Fe-Mn-W thin films are shown in figure 3(a), 3(b) and 3(c). Table 1 revealed that remanent polarization and coercivity values were increased with increase of current density and deposition time [4].

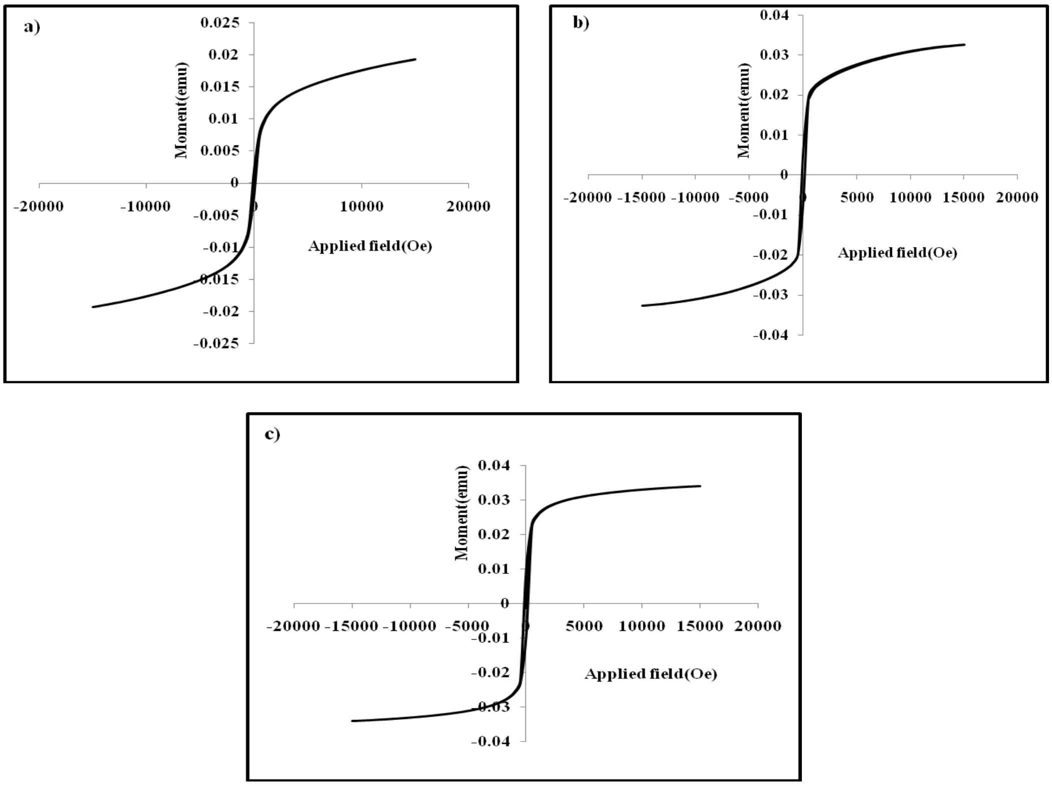

Fig. 3. VSM images of Fe-Mn-W films electrodeposited for 45 mins at a) 20 mA cm^{-2} b) 25 mA cm^{-2} c) 30 mA cm^{-2}

4. Conclusions

The Fe-Mn-W magnetic thin films were successfully prepared by electrodeposition method. From SEM analysis the films were obtained gray in colour with smooth, homogeneous surface at low current densities and also the films had good adhesion on the substrate. When current density was increased from 20 mA cm^{-2} to 30 mA cm^{-2}cracks were produced at 30 mA cm^{-2} due to internal stress in the film and also hardness values of the films increased. The XRD analysis revealed that the films have crystalline nature such as hexagonal and cubic structure and their crystalline sizes are in nano scale. The high remanent polarization, coercivity and squareness values were observed as 9.316 x 10^{-3} (emu), 147.67 Oe and 0.274 for the current density 30 mA cm^{-2} with 45 minute deposition time. The Fe-Mn-W thin films of magnetic, structural and mechanical properties which are used in various electronic devices such as recording media, magnetic magnetic shielding, magnetic actuators, writing heads, magnetic data storages and MEMS devices.

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