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SYNTHESIS AND CHARACTERIZATION OF Ni-Fe-S MAGNETIC THIN FILMS AT DIFFERENT BATH TEMPERATURES

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ABSTRACT

Electrodeposited alloy thin films Ni-Fe-S were prepared at temperatures 30, 50, 70 and 90˚C. Electrodeposited Ni-Fe-S thin films were subjected to morphological, structural, magnetic and mechanical characterization analysis. The chemical composition of the coated films was analyzed by EDAX and result shows that nickel content increases when bath temperature is increased. The surface and structural morphology of the coated film were analyzed by using SEM and XRD. The results show that thin films are bright, crack free and uniform. The mechanical characterization of Ni-Fe-S thin films have been analyzed by VHT and result shows that hardness increases when bath temperature is increased. The electroplated Ni-Fe-S thin films were strongly adherent to the copper substrate. All the electrodeposited Ni-Fe-S films exhibit FCC crystalline structure by XRD study.The VSM result of Ni-Fe-S thin films shows that thin films coated at high bath temperature have highest saturation magnetization value.

Keywords: Thin films, Characterization, Electrodeposition, Crystalline size, Temperature, X-ray diffraction, Microhardness, Surface morphology.

INTRODUCTION

Thin film technology is the foundation of all the astounding improvements made in solid state electronics. Electroplated Ni-Fe is one of the most widely used materials in the fabrication of micromachines such as microcantilevers, micro gears, and their components.[1-5] Electrochemical deposition or electrodeposition is one of the most suitable techniques employed in the preparation of thin films on the surface of conducting substrate. NiFe, NiCo, and NiW are most commonly used magnetic thin film materials in MEMS and NEMS.[1-3, 6-9] NiFe soft magnetic thin films with low coercive force and high permeability have been widely applied in the electromagnetic wave absorbing field. The electroplated thin films of Ni, Co, Fe metals have been developed by their potential applications in NEMS and MEMS.[1,10-12] The performance of NiFe soft film can be enhanced by adding stress reducing agent sulfur with NiFe alloy. In this current investigation, the electrodeposition method has been chosen for coating Ni-Fe-S thin films. This paper summarizes the preparation technique and characterization of electroplated Ni-Fe-S magnetic films.

EXPERIMENTAL

The different working conditions and bath composition of the Ni-Fe-S thin film are shown in Table-1.The Ni-Fe-S thin films were successfully coated by electrochemical deposition method at various bath temperatures such as 30, 50, 70 and 90^0C. The steel and copper substrates were used as anode and cathode respectively with the dimension of 7.5 x 1.5 cm. The cathode (copper substrate) was totally shielded with an adhesive tape except for the deposition area.[13-15,18] Before electrodeposition process, both anode and cathode substrates were polished and cleaned in NaOH solution. Then they were washed with distilled water and dried in air.[16,17,19-21] The bath was maintained at 6 pH value. The films were coated on the cathode surface at various temperatures from 30^0C to 90^0C. All the Ni-Fe-S thin films were coated at a constant current density of 3 mA/cm^2 and constant deposition time of 15minutes. The cathode was carefully removed from the bath after 15 minutes and dried for few minutes. The surface morphology of Ni-Fe-S alloy films

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was investigated with the help of Scanning Electron Microscope (SEM). The film composition and structural characters of thin films were measured by Energy Dispersive X-ray Analyser (EDAX) and X-ray diffraction (XRD) respectively.[22-25]Also, the hardness of Ni-Fe-S thin films was measured by Vickers Hardness Tester (VHT).The magnetic property of Ni-Fe-S thin film was measured by Vibrating Sample Magnetometer (VSM).

Table-1: Electroplating bath details of Ni-Fe-S thin films

S. No.	Name of the chemicals	(g/L)	Temperature (^{o}C)	Current Density	pH
1	Ferrous Sulphate	10	30,50,70,90	3 mA/cm^2	6
2	Nickel Sulphate	30			
3	Thiourea	15			
4	Ammonium Sulphate	40			
5	Citric acid	10			
6	Boric acid	10			

RESULTS AND DISCUSSION

Composition of Electrodeposited Thin Films

The chemical composition of the electroplated thin films was analyzed by EDAX spectrum. The EDAX data of thin films are shown in Table-2. The EDAX result shows that thin films prepared at higher temperature have less percentage of sulfur content. The highest ferrous content of 16.41wt% is obtained at the temperature of 50˚C. From EDAX data's, it is concluded that Nickel content increases by increasing the bath temperature. At bath temperature 90˚C, maximum Ni content of 76.36 wt% is obtained for Ni-Fe-S thin films. When bath temperature is increased, the weight percentage of sulfur decreases.

Table-2: Chemical composition of Ni-Fe-S thin films (EDAX)

S. No.	Temperature	Fe (% Wt)	Ni(% Wt)	S(% Wt)
1.	30˚C	13.86	61.38	24.76
2	50˚C	16.41	63.10	20.49
3	70˚C	12.28	72.06	15.66
4	90˚C	11.36	76.36	12.28

Morphological Observation

Figure-1 shows surface morphology of the electroplated Ni-Fe-S thin films with different temperatures (30, 50, 70 and 90^{0}C) and thin films are smooth and uniform. They are bright and crack free. Based on SEM analysis, it is concluded that formation of Ni-Fe-S thin films on a copper substrate is uniform in nature.

Structural Analysis

Figure-2 shows the XRD results of the deposits obtained at temperatures 30^{0}C, 50^{0}C, 70^{0}C and 90^{0}C. The occurrence of sharp peaks in XRD pattern of Ni-Fe-S thin films shows that the deposits are in crystalline nature. The size of crystals in deposits is calculated by the formula:

$$D = 0.954\ \lambda / \beta \cos\theta$$

Where θ is the Bragg's angle, λ is the wavelength of X-rays, β is FWHM of the diffraction peak at 2θ. XRD results of Ni-Fe-S films reveal the presence of FCC phase with (111), (200) and (220) diffraction peaks. The result shows that the crystal sizes of thin film deposits obtained by electrochemical deposition process are in nanoscale and average crystal size is around 26 nm.

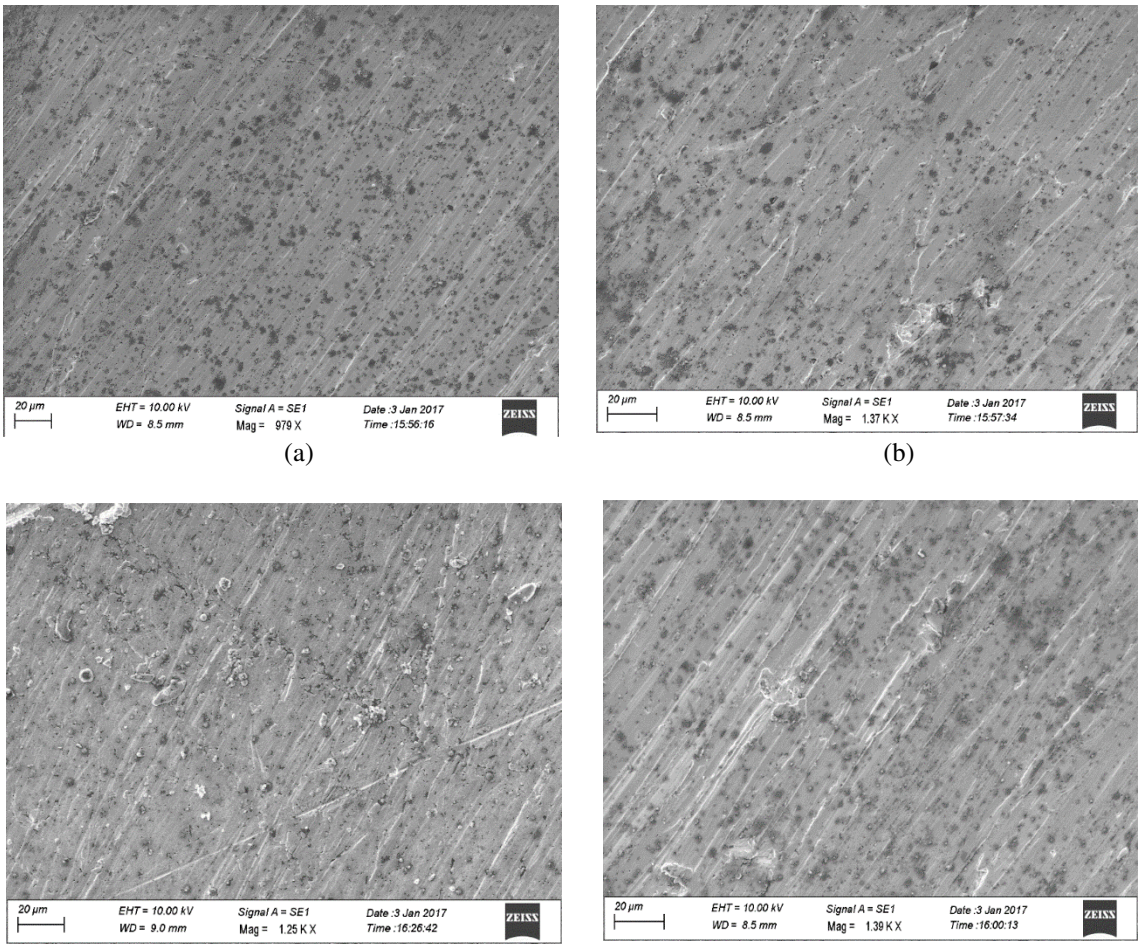
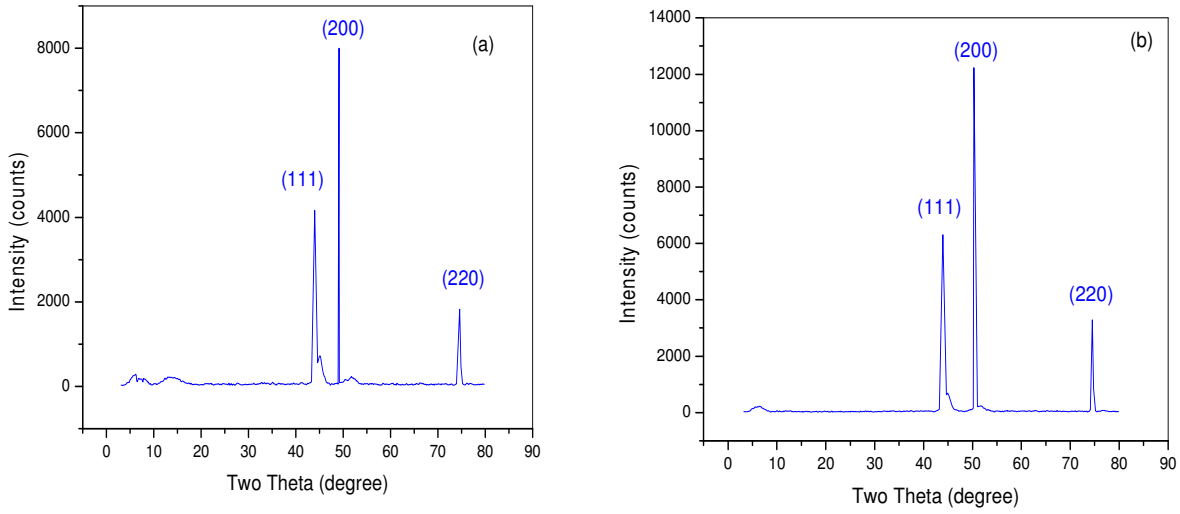


Fig.-1: Surface images (SEM) of Ni-Fe-S thin films at different bath temperatures
(a) 30˚C (b) 50˚C (c) 70˚C (d) 90˚C



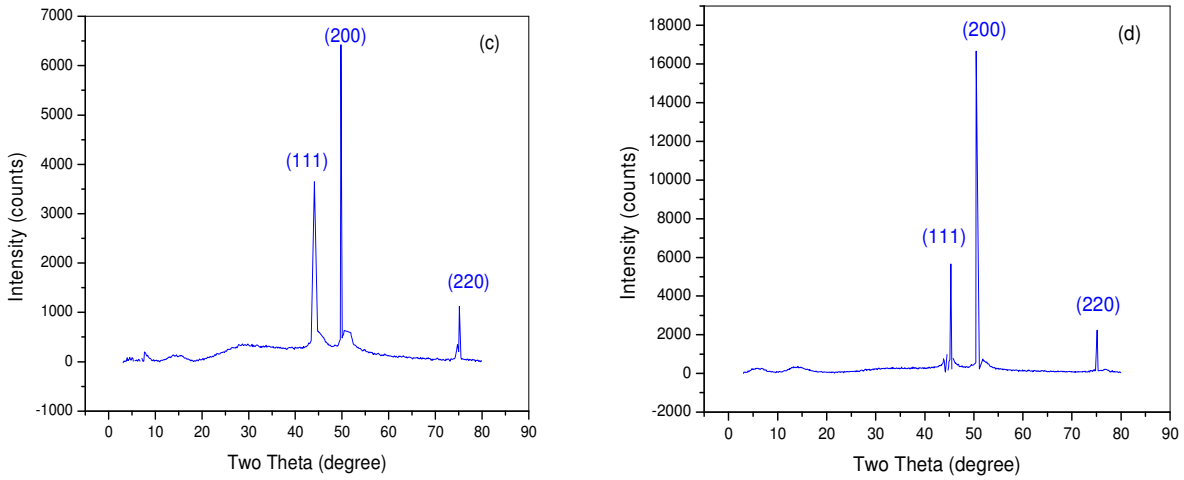


Fig.-2: XRD patterns of Ni-Fe-S thin films at (a) 30^0C (b) 50^0C (c) 70^0C (d) 90^0C

The crystal sizes of Ni-Fe-S alloy films are shown in Table-3. When bath temperature is increased, crystalline size of thin films decreases due to onset orientation of crystals during electrodeposition.

Table-3: Structural characteristics of Ni-Fe-S alloy thin films

S. No.	Bath Temperature (˚C)	2θ (deg)	d (A^0)	Particle Size(D) (nm)	Strain (10^{-3})	Dislocation Density (10^{14} / m^2)
1	30	49.16	1.8081	32.17	1.1254	09.66
2	50	50.32	1.8211	29.87	1.2121	11.21
3	70	49.71	1.8133	23.19	1.5612	18.60
4	90	50.58	1.7652	19.34	1.8720	26.74

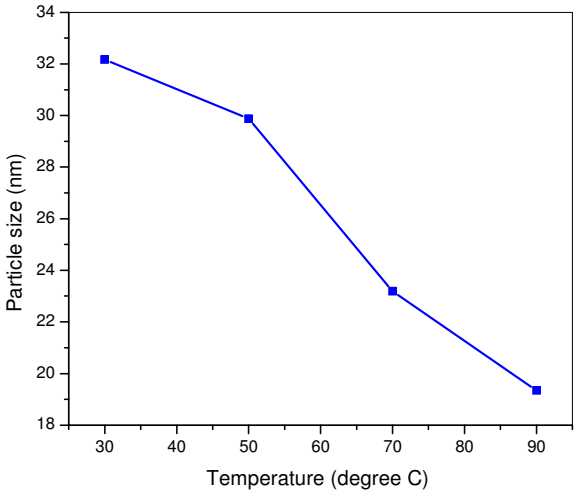


Fig.-3: Crystal size Vs Bath temperature

Mechanical Properties

Hardness is an important mechanical property of thin films. Vickers hardness tester is used to investigate hardness properties of thin films. VHT results show that the hardness of Ni-Fe-S increases when bath temperature is increased. Hardness increases due to the involvement of lower stress during formation of Ni-Fe-S films. The hardness of Ni-Fe-S alloy thin films is shown in Table-4.

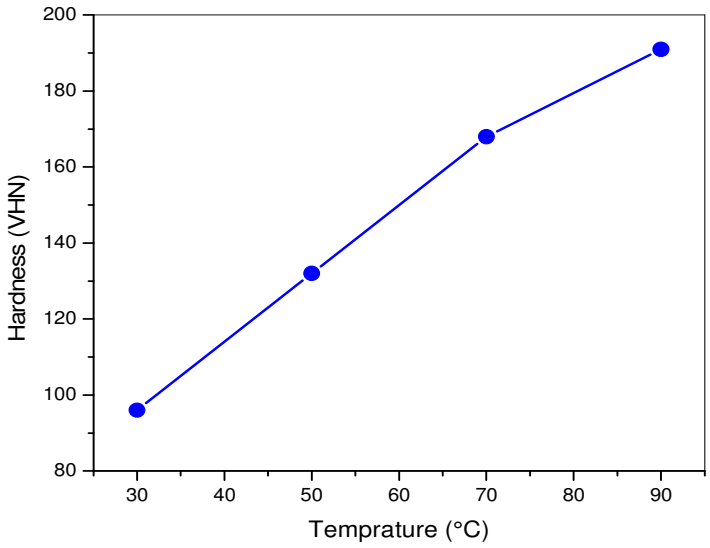


Fig.-4: Vickers Hardness Vs Bath temperature

Table-4: Hardness of Ni-Fe-S alloy thin films

S. No.	Bath Temperature(˚C)	Vickers Hardness(VHN)
1	30	96
2	50	132
3	70	168
4	90	191

Magnetic Properties of the Deposits

Magnetic properties of Ni-Fe-S films were observed by Vibrating Sample Magnetometer (VSM) and data's are shown in Table-5.The magnetic hysteresis curves of Ni-Fe-S thin films at different temperatures are shown in Figure-6.

Table-5: Soft Magnetic Properties of Ni-Fe-S deposits

S. No.	Bath Temperature (˚C)	Coercivity H_s (G)	Magnetization M_s (emu/cm^2) 10^{-3}	Retentivity M_r (emu/cm^2) 10^{-3}	Squareness $S(M_r/ M_s)$
1	30	241.32	7.327	5.7481	0.7845
2	50	334.26	13.623	3.2731	0.2402
3	70	352.73	77.271	32.725	0.4235
4	90	478.12	136.25	26.923	0.1976

Thin film coated at 90˚c of temperature reveals higher magnetization. It is observed that the magnetization increases from 7.327 x $\mathbf{10^{-3}}$ emu/cm^2 to 136.25 x $\mathbf{10^{-3}}$ emu/cm^2. So it is concluded that thin films prepared at high temperature (90˚C) exhibit higher values of saturation magnetization. A low coercivity is an essential character required for Ni based thin films. Grain size of the film plays a vital role in deciding coercivity of

thin film. If grain size is large, the properties of ferromagnetic materials are decided based on the domain wall movement. By reducing the grain size, magnetic properties of thin films can be changed. If grain size is in the range of nanometres, coercivity of magnetic materials is decreased. Coercivity of the films is also affected by other aspects such as impurities, film stress etc.

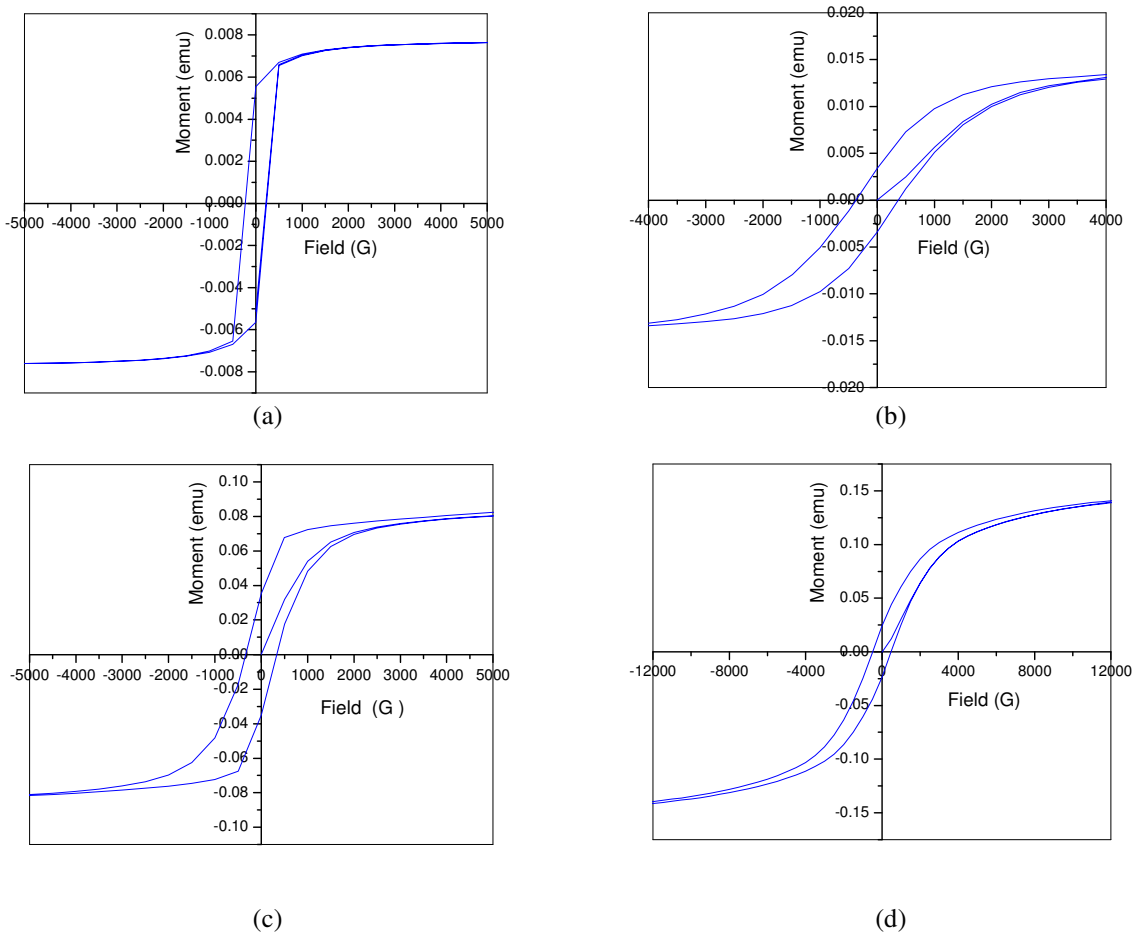


Fig.-5: Magnetic Hysteresis loops of Ni-Fe-S thin film at bath temperatures (a) 30˚C (b) 50˚C (c) 70˚C (d) 90˚C

Thin film stress is reduced because of temperature increases. Due to smaller crystalline size and low stress during formation of Ni-Fe-S thin films at 90˚C have higher saturation magnetization. By analyzing VSM results, it is concluded that the best soft magnetic thin films are obtained at high temperatures.

CONCLUSION

Electroplated Ni-Fe-S nanocrystalline thin films have been successfully coated on the copper substrate. The structural, mechanical and magnetic behaviors of Ni-Fe-S alloy thin films are analyzed. The result shows that the electroplated Ni-Fe-S alloy films have an average crystalline size of 26 nm and the films have FCC structure with three diffraction peaks. The thin films obtained at different temperatures are uniform, bright and crack free. While increasing the bath temperature from 30°C to 90°C, the Ni content of Ni-Fe-S thin increases and the Fe content decreases. The Ni-Fe-S thin films have good adherence with the substrate and Vickers hardness of the film is high as 191 VHN at 90°C. The Ni-Fe-S thin films coated at room temperature (30°C) have a coercivity of 241.32 Gauss with saturation magnetization value of 7.327 x 10^{-3} emu/cm^2.

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