SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF ELECTRO DEPOSITED NICKEL FERROUS SULFIDE ALLOY THIN FILM

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ABSTRACT

Nano crystalline Ni-Fe-S alloy thin films were deposited by electrodeposition method on the copper substrate at 30˚C .The structural, chemical composition, surface morphology and mechanical property of electro deposited Ni-Fe-S thin films were studied. EDAX investigation gives the chemical composition of the coated films. The structural character of the coated film has been analyzed by using XRD. Surface morphology of the coated film has been analyzed by using SEM. The mechanical properties of coated films were studied by Vickers hardness test.. By bend and scratch test, it was confirmed that electroplated Ni-Fe-S thin films were strongly adherent to the substrate The deposits of thin films were found to be smooth and nano crystalline. The coated Ni-Fe-S thin films exhibit the maximum Fe content of 65.21 wt%, Ni content of 29.52 wt% and 5.27 wt% of S. The Vickers hardness test shows 48 VHN while applying the load of 25g .Generally NiFe based thin films have potential applications in the fabrication of MEMS and NEMS devices. Based on the magnetic properties of thin films, this can be used in MEMS devices. The average crystalline size of Ni-Fe-S thin films were calculated from XRD is in range of 21.38 nm

Keywords: Thin Films, Characterization, Electrodeposition, Crystalline Size and Surface Morphology.

I. INTRODUCTION

Electrodeposited nanocrystalline Ni-Fe-S magnetic thin films have been developed due to their potential applications in power electronics, sensors, actuators, core material for writing elements in recording heads [1-4]. The Ni, Fe,Co, W, Cr based thin films have potential applications in the field of magnetic sensor technology, computer read/write heads, large scale integration (ULSI) devices, micro electro mechanical systems (MEMS) and Nano electro mechanical systems (NEMS) and based on their compositions, structural and magnetic properties. Electro deposited Perm alloy ($Ni_{80}Fe_{20}$) is the best known thin film alloy in magnetic thin film recording heads and MEMS applications [3]

In the current MEMS technologies NiFe FeS, CoW, NiS and NiW thin film alloys are used because those films can exhibit excellent magnetic properties [1-2].

The alloy thin films can be synthesized through several physical and chemical methods such as thermal decomposition method, spray pyrolysis and electrodeposition method. In this current investigation, the electrodeposition method has been chosen for coating the Ni-Fe-S thin films. The Low stress thin film alloys with improved magnetic properties are very much used in magnetic recording heads and MEMS [7-10].

The best known stress reducing agents for nickel based electrodeposition are sulfur containing organic additives (saccharin, thiourea, benzene sulfonic acid etc).Because compared to other coating methods electrodeposition has several advantages. The electrodeposition method is the simplest, most economical, reliable and reproducible technique. Electrodeposition of Ni-Fe films and their studies were carried out by few numbers of researchers [10-17].

Normally Ni-Fe thin films have good magnetic properties so that it can be used in various applications. If we add third element to FeNi the structural, mechanical and magnetic properties may be enhanced. In this present work we planned to analyze the effect of S on Ni-Fe thin films. This paper summarizes the synthesis and structural characterizations of Ni-Fe-S electroplated thin films.

II. EXPERIMENTAL PART

The bath composition of Ni-Fe-S alloy thin film is shown in Table 1.The thin films are successfully coated by electrodeposition method. In this investigation a copper plate of size 1.5 cm as breath and 7cm as length and same size of stainless steel were used as substrates.. Copper substrate act as the cathode and pure stainless steel act as the anode. Both cathode and anode were pre- treated by washing with soap and soaking in 20% H_2SO_4 for 3 minutes. Just before the deposition both the plates are degreased by acetone. The electroplating bath prepared by all the reagent grade chemicals was dissolved in triple distilled water.

The pH value of the bath was adjusted to 6 by adding few drops of ammonia solution. The Ni-Fe-S thin films were electro deposited on the copper substrate by applying a current of 7.5 mA for 15 minutes at room temperature. After 15 minutes the cathode was carefully removed from the bath and dried for few minutes. The mask on the surface of cathode was also given by using adherent tape. The structure and morphology of the Ni-Fe-S thin films were studied with the help of XRD and SEM.

The film composition was measured by Energy-dispersive X-ray Spectroscopy (EDAX).Hardness of the film was measured by Vickers Hardness Test (VHN).The thicknesses of the films were determined by cross sectional view of SEM images. The experimental setup of electrodeposited Ni-Fe-S thin films is shown in fig 1.

Table 1: Electrode Position Bath Details of Thin Films

S. No	Name of the chemicals and parameters	Data g/1
1.	Nickel Sulphate	30
2.	Ferrous Sulphate	15
3.	Thiourea	10
4.	Ammonium Sulphate	40
6.	Boric acid	20
7.	pH value	6
8.	Time Duration	20 min
9.	Current density	1 mA/cm^2

Figure 1: Experimental Setup of Electrodeposited Ni-Fe-S Thin Films

III. RESULT AND DISCUSSION

3.1 Composition of the Electro Deposited Thin Films

The chemical composition of the electroplated thin films has been analyzed by EDAX spectrum. The EDAX spectrum of Ni-Fe-S thin films is shown in fig 2. The EDAX data of thin films are shown in Table 2.

Table 2: EDAX analysis of thin films

S. No	Temperature	Fe Wt%	Ni Wt%	S Wt%
1.	Room Temperature	65.21	29.52	5.27

From EDAX, we conclude that, the electroplated thin films have maximum Fe content of 65.21 wt% , 29.52 wt% of Ni and 5.27 wt% of S.

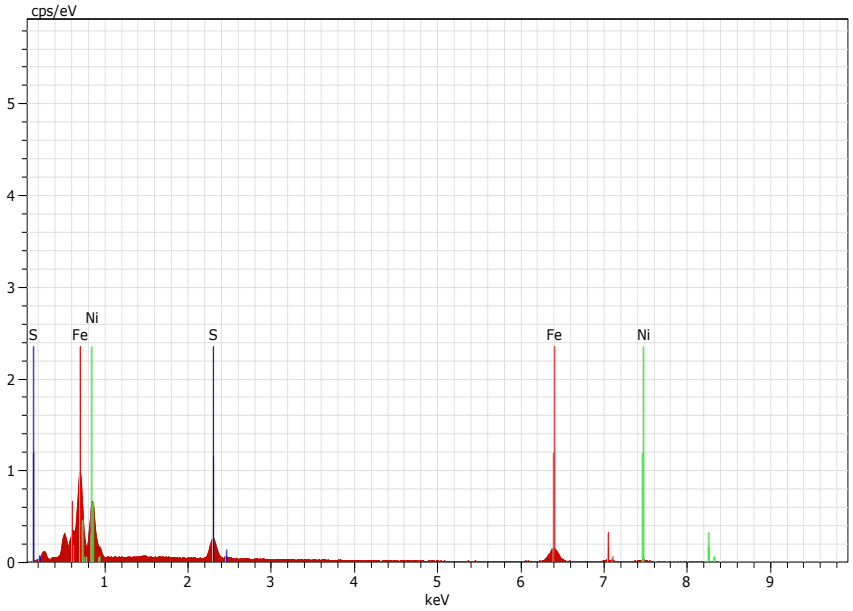

Figure 2: EDAX Spectrum of Thin Film

3.2 Surface Morphology of Thin Films

The surface morphology of the electroplated Ni-Fe-S thin films is analyzed by using SEM pictures and are shown in fig 3.The electroplated thin films are smooth, uniform and adherent with substrate. The thin films are crack free and uniform. From SEM analysis we conclude that the formation of Ni-Fe-S thin films on the copper substrate is uniform in nature. The thickness of the coated thin films is 5.8 μm determined from cross sectional view of SEM images.

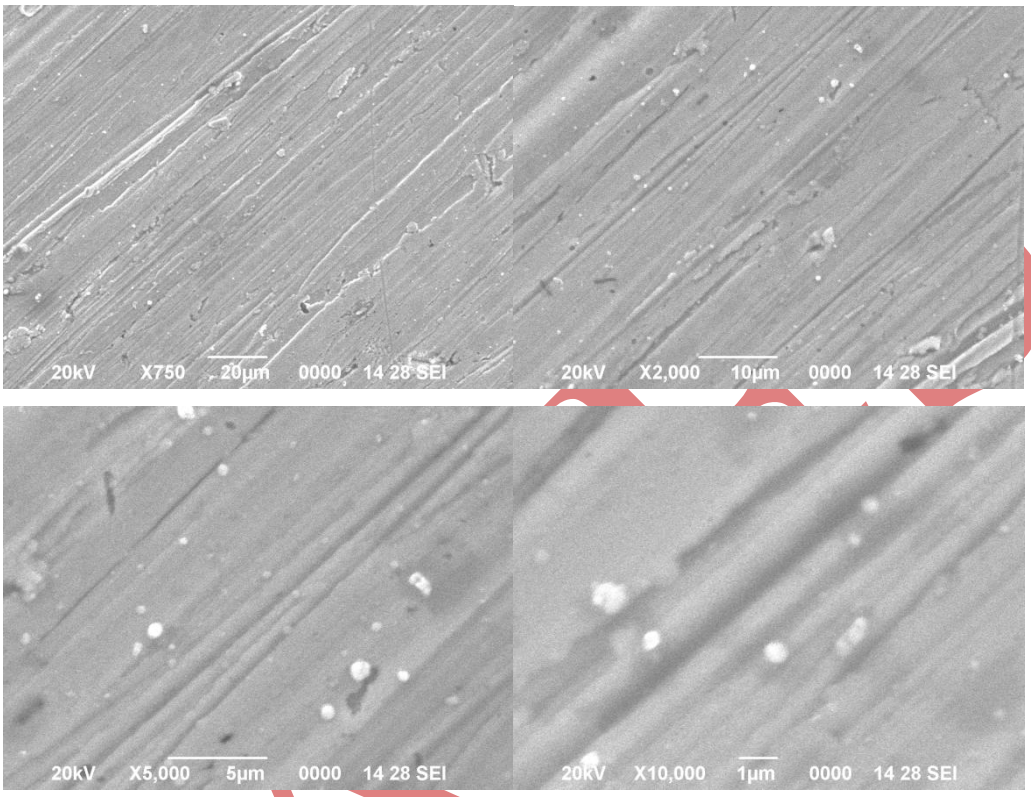

Figure 3: SEM Images of Electrodeposited Thin Films

3.3 Structural Properties of Ni-Co-B Thim Films

The crystal structure of the electro deposited Ni-Fe-S alloy thin films were determined by XRD analysis. X- ray diffraction patterns of films obtained at room temperature were shown in fig 4. The presence of sharp peaks in XRD pattern reveals that the films are crystalline in nature. Crystalline size of the deposits of Ni-Fe-S thin films were calculated from XRD pattern using formula

$$(D=0.954\lambda/\beta\cos\theta) \text{ ----------(1)}$$

The strain (ε) was calculated from the relation

$$\varepsilon = \frac{\beta \cos\theta}{4} \text{ ---------- (2)}$$

The data's obtained from XRD analysis of Ni-Fe-S thin film are compared with standard JCPDS data and found to have FCC crystalline structure with three predominant peaks of (200), (111) , (220) . These values clearly show that the crystalline sizes of the deposits obtained by electro deposition process are in the nano scale. The crystal size of alloy films are tabulated as shown in Table 3. The average crystalline size of the thin films are around 21.38 nm.

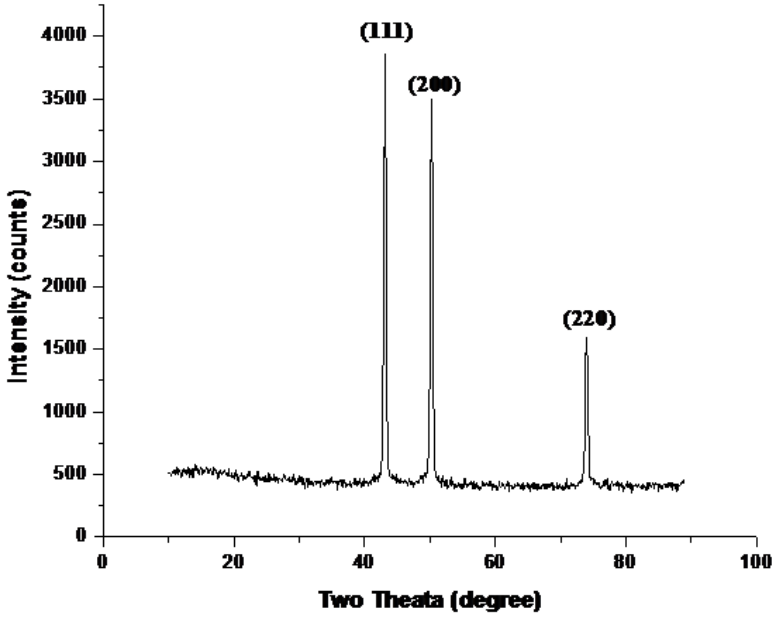

Figure 4: XRD Pattern of Electrodeposited Thin Film

From XRD we conclude that the films have nano crystalline phase. Because Nano scale level is the dominant factor to decide the magnetic properties. If we increase the bath temperature the crystalline size of Ni-Fe-S thin films may be decreased due to onset orientation of crystals during electrodeposition. So we planned to investigate the magnetic properties of Ni-Fe-S thin films with different bath temperature and different sulfur concentration.

Temperature (^{0}C)	2Θ (deg)	d (A^0)	Particle size, D (nm)	Strain (10^{-3})	Dislocation density (10^{14} / m^2)	Thickness (µm)
30	43.07	2.0982	20.18	1.8052	24.5618	5.8
	50.9	1.8160	21.34	1.7066	21.9518	
	73.92	1.2810	22.62	1.6103	19.5443	

Table 3: Crystalline Size of Alloy Thin Film

3.4 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. Initially the equal lines are drawn on the surface of the coated film by pin. After that the adhesive tape is pasted over the coated surface and then finally pulls out the tape from the surface of coated Ni-Fe-S thin films. If the film comes with tape then the adhesion is poor. This test showed that the film is having good adhesion with the substrate. Hardness of the films was examined by using a Vickers hardness tester by the diamond intender method. Vickers hardness test value is low in the order of 48 VHN while applying the Load of 25g.

IV. CONCLUSION

The Ni-Fe-S magnetic thin films were successfully synthesized by electro deposition at room temperature. The nano crystalline films obtained at room temperature are crack free, bright and uniform. FCC was the dominant structure of electro deposited Ni-Fe-S thin films. The crystalline sizes of the deposits obtained by electro deposition process are in the nano scale. The average crystalline size of Ni-Fe-S films is around 21.38 nm. Hardness of this magnetic thin film is 48 VHN. Based on their magnetic properties of thin films, these films can be used in various electronic devices including high density recording media, magnetic actuators, magnetic shielding, magnetic writing heads, high performance transformer cores, MEMS and NEMS. Because of the potential applications of FeNi based thin films in various industrial areas we planned to analyze the magnetic property of thin films in our future research work.

REFRENCES

[1] Senoy Thomas, SH Al-Harthi, D.Sakthikumar IA.Al-omari, RV Ramanujam, Yasuhiko Yoshida and MR Anantharaman, J.Phys.D:Appl.phys, **41,** 155009(8pp), (2008).

[2] Daheum Kim, D.-Y Park, B.-Y.Yoo, P.T.A.Sumodjo, N.V.Myung, Electrochimica Acta, **48,** 819-830 (2003).

[3] Tetsuya Osaka, Toru Asahi, Jun Kawaji, TokihikoYokoshima, ElectrochimicaActa, **50**, 4576-4585, (2005).

[4] M. Zielinski, E. Miekos, J Appl Electrochem **38,** 1771 (2008).

[5] Amaresh Chandra Mishra, Physica B, **407**, 923-934, (2012).

[6] D.Niarchos, Sensors and Actuators A, **106**, 255-262, (2003).

[7] L.X.Phua, N.N. Phuoc, C.K. .Ong, Journal of alloys and Compounds, **520,** 132-139, (2012).

[8] Nosang V.Myung, D.-Y Park, B.-Y.Yoo, Paulo T.A.Sumodjo, J. of Magn and Mag Mat, **265,** 189-198(2003).

[9] M.Jayalakshmi, Woo-Young Kim, Kwang-Deog Jung, Oh-ShimJoo, Int.J, Electrochem.Sci. **3**, 908-917, (2008).

[10] H.Gavrila, V.Ionita, J.optoelectronics and advanced materials, **4**, No 2, P.173-192 (2002).

[11] N.Sulztanu, Fbrinza, J. of Optoelectronics and Advanced Materials, **6**, No-2, P.641-645 (2004).

[12] R. Kannan, S. Ganesan, T. M. Selvakumari, Optoelectron. Adv. Mater. – Rapid Commun., **6**(3-4), 383 (2012).

[13] T.Baskar,K.S.Rajni,R.Kannan,Synthesis and Structural Characterization of electrodeposited CoNiP alloy thin film,IJATES,vol. no-2(2014)

[14] B. Tury, M.Lakatos-Varsanyi and S. Roya, "Ni-Co Alloys Plated by Pulse Currents," *Surface & CoatingsTechnlogy, Vol, pp.6713-6717*,**(2006).**

[15] T.Baskar,K.S.Rajni,R.Kannan,Synthesis and Structural Characterization of electrodeposited Nickel Cobalt Boron alloy thin film,IJARSE,vol. no-3(2014)

[16] R.Kannan, S.Ganesan, T.M.Selvakumari, Digest J. of Nanomaterials and Biostructures, **7**, No3, 1039-1050, (2012).

[17] T. M. Selvakumari, P. Muthukumar, S.Ganesan, Digest J. of Nanomaterials and Biostructures, **5(**4), 903 (2010).