Effect of Annealing and Properties of Electrodeposited Fe-Ni-Co Thin Films

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***Abstract*- Nano crystalline FeNiCo alloy thin films with annealingis deposited on the copper substrate by electro deposition method. Electro deposited FeNiCo thin films with before and after annealing are subjected to the structural, morphological and mechanical characterization analysis. The chemical composition of the coated thin films is analysed by EDAX. The surface and structural morphology of the coated film are analysed by SEM and XRD. The mechanical properties of FeNiCo thin films are analysed by VHT. The SEM pictures of FeNiCo thin films show that the deposits of thin films are crack free, uniform and bright surface. All the electro deposited FeNiCo films exhibit FCC crystalline structure. The VHN result of FeNiCo thin films shows that thin films coated at high bath temperature have highest saturation hardness value. Fe-Ni-Co thin films can be used for the manufacturing of MEMS and NEMS devices.**

***Keywords* - Thin films, Characterization, Electrodeposition, Crystalline size, Temperature, X-ray diffraction, Micro hardness, Surface morphology**

I. INTRODUCTION

The importance of electro deposition as a fabrication technology in the electronic industry is large and growing [1-3]. Electro deposited magnetic thin films are important in computer read/write heads and MEMS because of their fiexibility, capability, quality and low cost. The most commonly used magnetic materials in MEMS and NEMS are soft magnetic materials, such as NiFe, NiCo and NiW [4-6]. The electro deposition technique is especially interesting due to itscost-effectiveness, easy maintenance and quality deposits. The combination of good mechanical and structural properties lead to the use of electroplated NiFe films in actuators, microscopic sensors, micro motors and frictionless micro gears [7-9]. The use ofNiFe as the soft film which can be improved by adding a third element with NiFe alloy. Permalloy[NiFe] is the best known thin film alloy in MEMS applications [10-13]. In this current investigation, the electro deposition method is selected for coating FeNiCo thin films. In this present work, it is analysed that the effect of different concentration of cobalt on FeNiCo thin films. This paper summarizes the synthesis and characterizations of electroplated FeNiCo thin films with different concentration of cobalt.

II. EXPERIMENTAL PART

The working conditions and bath composition of FeNiCo alloy thin film are shown in Table 1.The FeNiCo thin films are successfully coated by electro deposition method. In this investigation, Copper and Stainless steel substrates act as cathode and anode respectively. A copper plate and stainless steel of size 1.5 cm as breath and 7.5 cm as length are used as substrates. The FeNiCo thin films are electro deposited on the copper substrate by applying a current of 2 mA for 15 minutes with Nickel Sulphate 30 grams /Lit, Ferrous Sulphate 15 grams/Lit and Cobalt Sulphate 25 grams/Lit at 30˚C. The cathode is carefully removed from the bath after 15 minutes and dried for few minutes. The surface morphology of the FeNiCo thin films is analysed with the help of Scanning Electron Microscope (SEM). The film composition and structural characters of thin films are measured by Energy-dispersive X-ray Spectroscopy (EDAX) and X-ray diffraction (XRD) respectively. The hardness of Fe-Ni-Co thin films is measured by Vickers Hardness Test (VHN). The magnetic property of Fe-Ni-Co thin films is measured by Vibrating Sample Magnetometer (VSM). The thickness of the films are determined by cross sectional view of SEM images. The electro deposition bath details of FeNiCo thin films are given in Fig 1.

S.No	Name of the chemicals	Data (grams/Lit)
1	Nickel Sulphate	30
2	Ferrous Sulphate	15
3	Cobalt Sulphate	25
4	Ammonium Sulphate	40
5	Citric acid	10
6	Boric acid	10
7	Time Duration	15 min
8	Temperature	30˚C
9	Current density	2 mA/cm^2

Fig -1. Electroplating Bath Details of FeNiCo Thin Films

III. RESULTS AND DISCUSSION

A. Composition of Electro Deposited Thin Films

EDAX result shows that the films obtain with higher concentration of Cobalt Sulphate have high cobalt content. The highest cobalt content of 79.24 wt % is obtained with high Cobalt Sulphate concentration. EDAX result shows that Ni content decreased with decreasing the Cobalt Sulphate concentration. The maximum Ni content of 19.87wt % is obtained for FeNiCo thin films with low Cobalt Sulphate

concentration. The weight percentage of Co increased while increasing the Cobalt

Sulphate concentration. Ammonia solution is used to correct the pH value of the bath solution and its effect on the film is ignored.

Chemical Composition of FeNiCo Thin Films (EDAX)

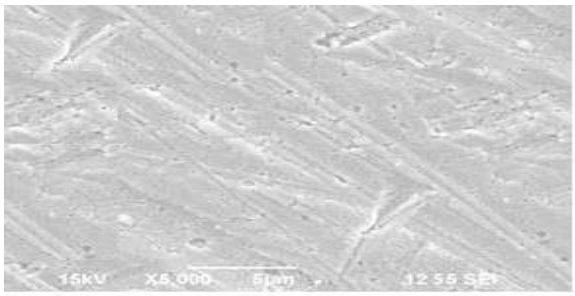
S. No	Condition	Co Wt%	Ni Wt%	Fe Wt%
1.	Before Annealing	68.45	19.87	11.68
2.	After Annealing	70.14	18.71	11.15

Fig -2: EDAX Analysis of Thin Films

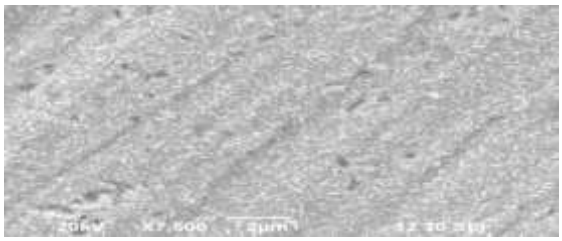
B. Morphological Observation

The surface morphology of the electroplated Fe-Ni-Co thin films with different concentration of Cobalt Sulphate is analysed by SEM pictures and are shown in Figure 3. The electroplated thin films are smooth and uniform. The thin films are bright, crack free and uniform. From SEM analysis it is concluded that the formation of thin films on the copper substrate is uniform in nature.

(a) 15 grams/lit



(b) 25 grams/lit



(c) 35 grams/lit

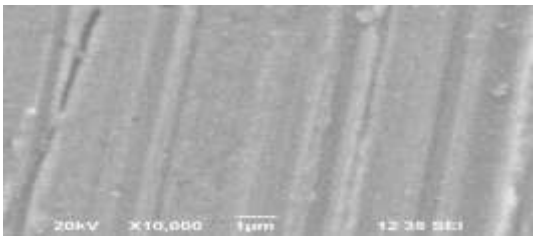


Fig -3: SEM Images for Electrodeposited Fe-Ni-Co thin films for different concentration of Cobalt Sulphate

C. Structural Analysis

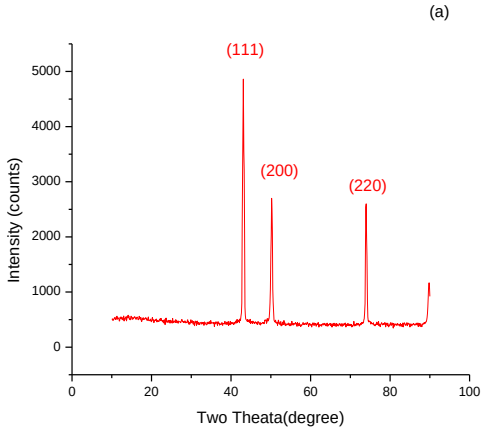
The crystal structure of the electrodeposited Fe-Ni-Co alloy thin films is determined by XRD analysis. X- ray diffraction patterns of Fe-Ni-Co films obtain with different concentration of Cobalt Sulphate are shown in Fig 4. The presence of sharp peaks in XRD pattern reveals that the films are crystalline in nature. The crystalline size of the deposits is calculated from XRD using Scherrer's formula

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

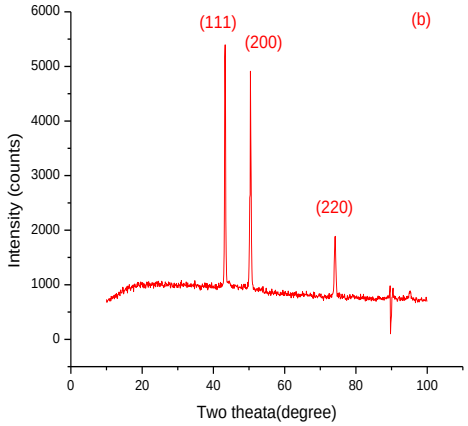
Where, θ is the Bragg's angle, λ is the X-ray wavelength, β is the full width at half maximum intensity of the diffraction peak located at 2θ. The XRD patterns of NiCoFe films reveal the existence of FCC phase with (111), (200) and (220) diffraction peaks. The result shows that the crystalline sizes of the Fe-Ni-Co deposits are obtained by electro deposition process in the nano scale and the average crystallite size is around 21 nm.

Structural Study of FeNiCo Thin Films (XRD)

(a) 15 grams/lit



(b) 25 grams/lit



(c) 35 grams/lit

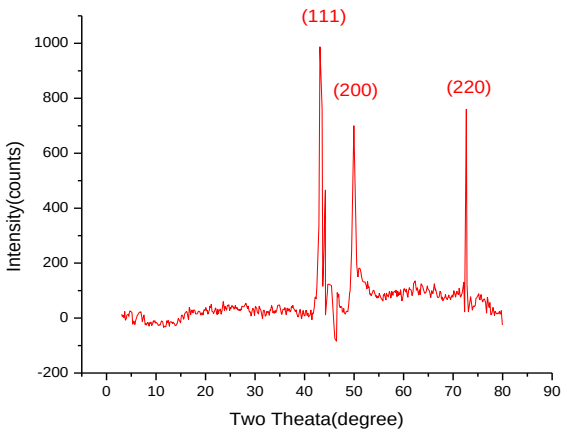


Fig-4 XRD pattern of electro deposited FeNiCo thin films with different concentration of Cobalt Sulphate.

The crystal size of Fe-Ni-Co alloy films is tabulated and shown in Fig5. When the concentration of Cobalt Sulphate is increased, the crystalline size of thin films is decreased due to onset orientation of crystals during electro deposition.

S. No	Cobalt Sulphate (grams /Lit)	2Ɵ (deg)	d (Å)	Particle size, D (nm)	Strain (10^{-3})	Dislocation density (10^{14} / m^2)
1	15	43.310	1.5634	23.32	1.578	21.87
2	25	44.528	1.3745	26.62	1.845	24.23
3	35	42.223	1.6235	27.23	1.429	22.17

Fig 5: Structural Characteristics of FeNiCo Alloy Thin Films

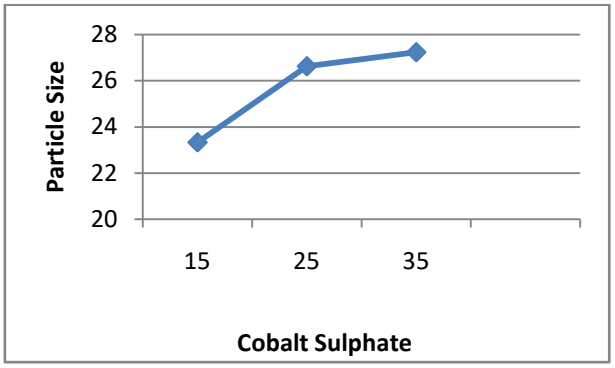


Fig 6 :Crystalline Size as a function of concentration of Cobalt Sulphate

D. Mechanical Properties

Hardness of the films is examined by Vickers Hardness tester. The results show that the hardness reduced when concentrationof Cobalt Sulphate in increased. This may happen due to lower stress associated with electro deposited Fe-Ni-Co films. The hardness of Fe-Ni-Co thin films are been shown in Fig 7.

S.No	Cobalt Sulphate (grams/Lit)	Vickers Hardness (VHN)
1	15	76
2	25	63
3	35	56

Fig7.: Mechanical Properties of Electrodeposited Ni-Co-Fe Thin Films

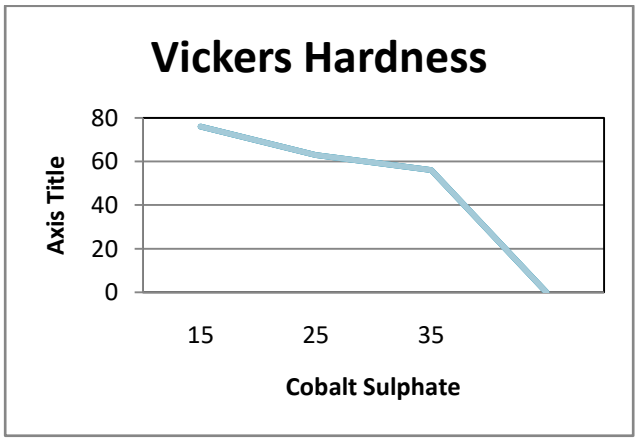


Fig-8: Vickers Hardness as a function of concentration of Cobalt Sulphate

E. Magnetic Properties of FeNiCo Thin Films (VSM)

The magnetic properties of the electro deposited FeNiCo films are analyzed by VSM. The magnetic hysteresis loops of FeNiCo alloy thin films for different temperatures are shown in Fig. 10

It is noted that the magnetization increases from 2.4703 $\times 10^{-3}$ emu/cm^2 to 4.2874$\times 10^{-3}$ emu/cm^2 when concentration of ferrous increases from 15 gm to 35 gm. It is concluded that the films prepared by 30 gm exhibits a lower value of saturation magnetization but Coercivity increases.

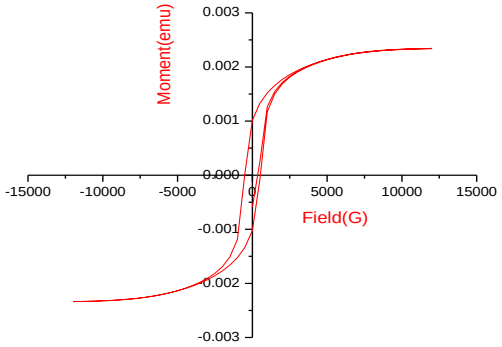
If the grain size is in the range of nanometers, coercivity of magnetic materials will decrease (grain size is smaller than the domain wall width). Also the coercivity of the films is affected by factors such as film stress, impurities etc.

S.No	Cobalt Sulphate (grams)	Coercivity H_s(G)	Magnetization M_s(emu/cm^2) $\times 10^{-3}$	Retentivity M_r(emu/cm^2) $\times 10^{-3}$
1	15	570.27	2.4703	1.0534
2	25	721.21	1.7934	0.9376
3	35	767.64	1.16731	0.78245

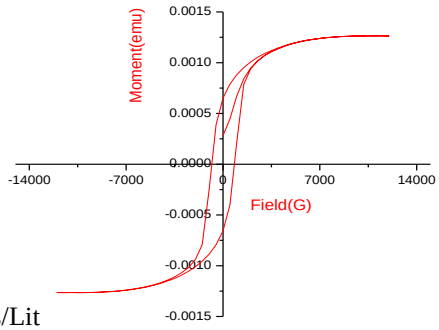
Fig 9. Magnetic Properties

Magnetic Hysteresis Loops

a) 15 grams/Lit



(b) 25 grams/Lit



(c) 35 grams/Lit

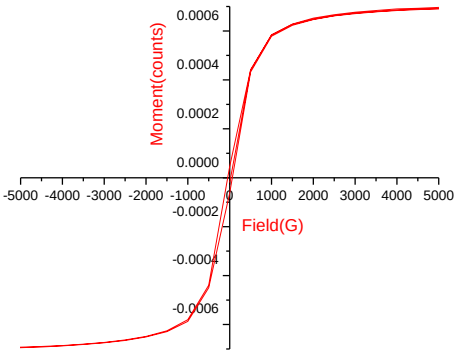


Fig 10.Magnetic hysteresis loops of FeNiCo thin film prepared with different Cobalt concentration (a) 15 grams/Lit (b) 25 grams/Lit (c) 35 grams/Lit

IV. CONCLUSION

The Ni-Co-Fealloy thin films are successfully synthesized by electro deposition with different concentration of Cobalt Sulphate. The nano crystalline films are obtained with different concentration of Cobalt Sulphate are crack free, bright and uniform. FCC is the dominant structure of electrodeposited Ni-Co-Fe thin films. The crystalline sizes of the deposits are in the nano scale. Hardness is increased with increasing concentration of Cobalt Sulphate.When the concentration of Cobalt Sulphate is increased from 10 g to 30 g, the particle size values decrease from 23.32 nm to 18.96 nm. This happens due to nano crystalline structure and low film stress associated with Fe-Ni-Co. This article summaries the optimized operating condition of electroplated bath. The Ni-Co-Fe thin films can be used in various electronic devices including high density recording media, magnetic writing heads, high performance transformer cores, MEMS and NEMS.

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