

EFFECT OF DIFFERENT FERROUS CONCENTRATION ON STRUCTURAL AND MECHANICAL PROPERTIES OF ELECTRODEPOSITED FeNiCo THIN FILMS

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ABSTRACT

Nano crystalline FeNiCo alloy thin films with different concentration of ferrous are deposited on the copper substrate by electrodeposition method. Electrodeposited FeNiCo thin films with different concentration of ferrous 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 electrodeposition as a fabrication technology in the electronic industry is large and growing (1-3). Electrodeposited magnetic thin films are important in computer read/write heads and MEMS because of their flexibility, 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 electrodeposition technique is especially interesting due to its cost-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, micromotors and frictionless microgears (7-9). The use of NiFe 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 electrodeposition method is chosen for coating FeNiCo

thin films. In this present work, it is analysed that the effect of different concentration of ferrous on FeNiCo thin films. This paper summarizes the synthesis and characterizations of electroplated FeNiCo thin films with different concentration of ferrous.

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 electrodeposition 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 breadth 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 15 mA for 15 minutes and varying the ferrous concentration (10, 20, 30 g/lit of ferrous) 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 thicknesses of the films are determined by cross sectional view of SEM images. The electrodeposition bath details of FeNiCo thin films are given in table 1.

Table 1. Electroplating bath details of FeNiCo thin films

S.No	Name of the chemicals	Data (g/L)
1	Nickel Sulphate	30
2	Cobalt Sulphate	15
3	Ferrous Sulphate	10,20,30
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

III. RESULTS AND DISCUSSION

3.1 COMPOSITION OF ELECTRODEPOSITED THIN FILMS

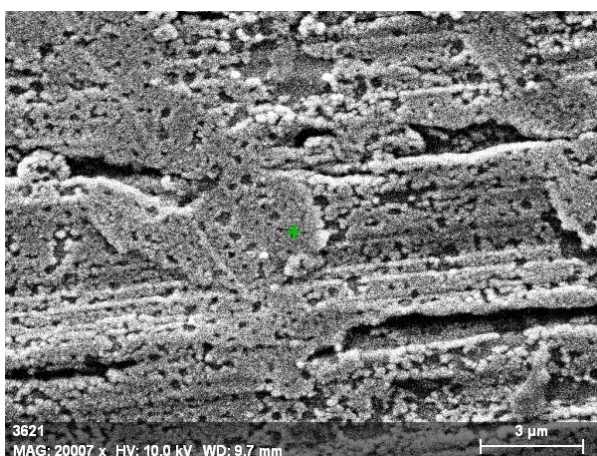
EDAX result shows that the films obtain with higher concentration of Ferrous Sulphate have high ferrous content. The highest ferrous content of 34.45 wt% is obtained with high Ferrous Sulphate concentration. EDAX result shows that Ni content increases with increasing the Ferrous Sulphate concentration. The maximum Ni content of 46.21 wt% is obtained for FeNiCo thin films with high Ferrous Sulphate concentration. The weight percentage of Co decreases while increasing the Ferrous Sulphate concentration. Ammonia solution is used to correct the pH value of the bath solution only and its effect on the film is ignored.

Table 2: EDAX analysis of thin films

S. No	Ferrous Sulphate(g)	Co Wt%	Ni Wt%	Fe Wt%
1.	10	68.45	19.87	11.68
2	20	41.57	31.11	27.32
3	30	19.34	46.21	34.45

3.2 MORPHOLOGICAL OBSERVATION

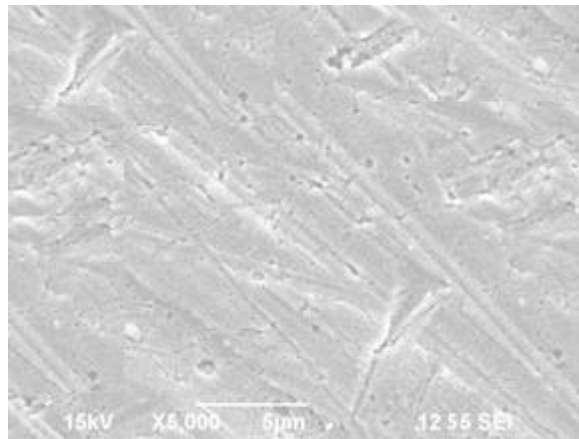
The surface morphology of the electroplated Fe-Ni-Co thin films with different concentration of Ferrous Sulphate is analysed by SEM pictures and are shown in fig 1. The electroplated thin films are smooth and uniform. The thin films are bright, crack free and uniform. From SEM analysis it is concluded that the formation of thin films on the copper substrate is uniform in nature



(a)



(b)



(c)

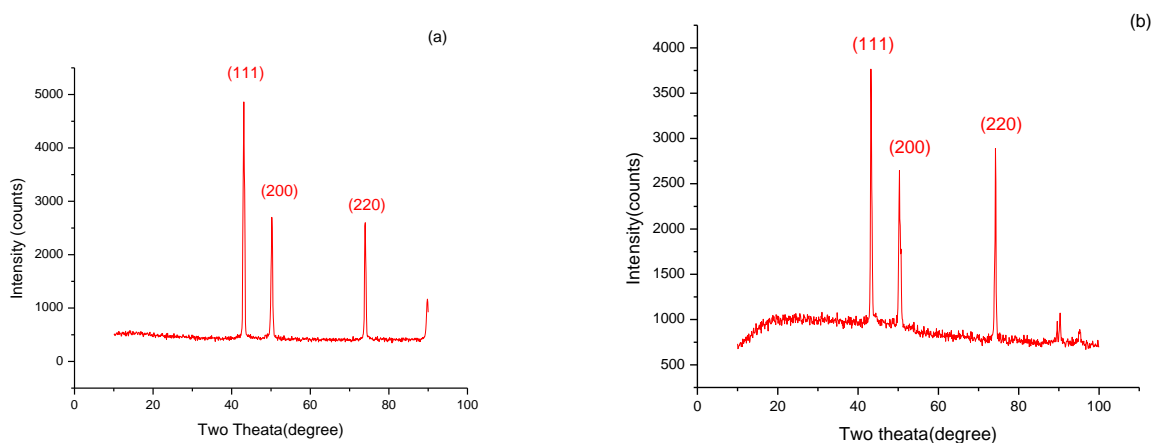
Figure 1: SEM Images for Electrodeposited Fe-Ni-Co thin films for different concentration of Ferrous Sulphate (a) 10 g/l (b) 20 g/l (c) 30 g/l

3.3 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 obtained with different concentration of Ferrous Sulphate are shown in fig 3. 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 FeNiCo 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 electrodeposition process in the nano scale and the average crystallite size is around 21 nm.



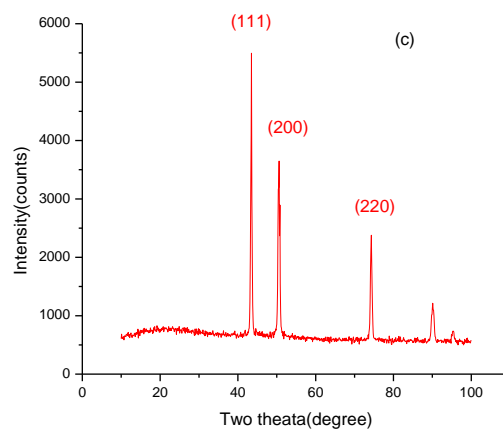


Fig.2 XRD Patterns of Fe-Ni-Co thin films for different concentration of Ferrous Sulphate (a) 10 g/l (b) 20 g/l (c) 30 g/l

The crystal size of Fe-Ni-Co alloy films is tabulated and shown in table 3. When the concentration of Ferrous Sulphate is increased, the crystalline size of thin films is decreased due to onset orientation of crystals during electrodeposition

Table.3 : Structural characteristics of FeNiCo alloy thin films

S. No	Ferrous Sulphate(g)	2 θ (deg)	d (\AA)	Particle size, D (nm)	Strain (10^{-3})	Dislocation density ($10^{14} / \text{m}^2$)
1	10	43.310	1.5634	23.32	1.578	21.87
2	20	42.932	1.4131	21.20	1.463	20.62
3	30	42.881	1.3215	18.96	1.612	18.07

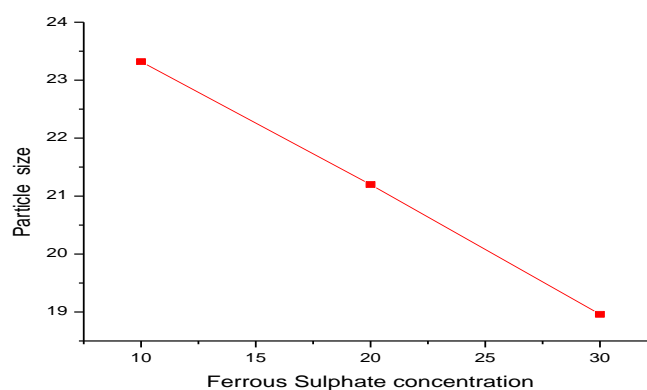


Figure 3. Crystalline Size as a function of concentration of Ferrous Sulphate

3.4 MECHANICAL PROPERTIES

Hardness of the films is examined by Vickers Hardness Tester. The results show that the hardness increases with increasing concentration of Ferrous Sulphate. This may happen due to lower stress associated with electrodeposited Fe-Ni-Co films. The hardness of Fe-Ni-Co thin films are shown in table 4.

Table.4: Mechanical Properties of electro deposited Ni-Co-Fe thin films

S.No	Ferrous Sulphate (g)	Vickers Hardness (VHN)
1	10	76
2	20	106
3	30	118

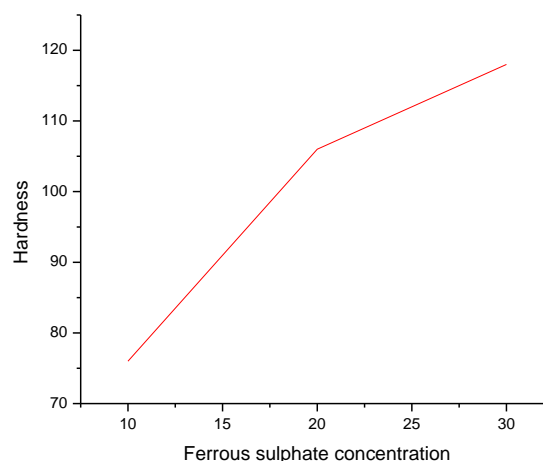


Figure 4. Vickers Hardness as a function of concentration of Ferrous Sulphate

IV. CONCLUSION

The Ni-Co-Fe alloy thin films are successfully synthesized by electro deposition with different concentration of Ferrous Sulphate. The nano crystalline films are obtained with different concentration of Ferrous 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 Ferrous Sulphate. When the concentration of Ferrous Sulphate is increased from 10 g to 30 g, the particle size values decreases 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.

**REFERENCES**

1. Brenner, "Electrodeposition of alloys Principle and practices", Vol. I and II, Academic Press, NewYork, 1963.
2. Damujanovic, A. "Modern aspects of Electrochemistry", Butterworths publishers Ltd., London, 1964.
3. Parthasaradhy, N.V. "Practical Electroplating Handbook", Prentice Hall, New Jersey, 1988.
4. Osaka, T "A soft magnetic CoNiFe film with high saturation magnetic flux density and low coercivity", Nature, 1998, 392, 796 – 798.
5. Ho Soon Min., "Metal Selenide semiconductor thin films: A Review", International Journal of ChemTech Research 2016, 9, 390-395.
6. Baskar.T, Rajni.K.S, "Effect of bath temperature on structural and magnetic properties of electrodeposited NiCoS magnetic thin films", International Journal of ChemTech Research, 2015,8, 234-239.
7. Baskar.T, Rajni.K.S, "Effect of different Sulfur concentration on structural and magnetic properties of electrodeposited NiCoS magnetic thin films", International Journal of ChemTech Research, 2016,5 ,317-324.
8. Thangaraj.N, Tamilarasan.K ,Sasikumar.D., "Effect of Phosphorous Acid on the Ferrous Tungsten Phosphorous Magnetic Thin Film", International Journal of ChemTech Research 2014, 6, 384-390.
9. Kavitha.N, Manohar.P., "Magnetic and Dielectric studies of Ni-Co-Zn Ferrites synthesized by Nonconventional combustion method", International Journal of ChemTech Research 2015, 8, 308-315.
10. Kannan, R, Kanagaraj, R &Ganesan, S 2013 'Influence of Tri Sodium Citrate Bath Concentration on the Electrodeposition of NiFeWS Thin Films', Journal of Ovonic Research, 2013,9, 45-54.
11. Iwasaki S., Nakamura Y., "An analysis for the magnetization mode for high density magnetic recording", Journal of Magnetism and Magnetic Materials., 1977, 200, 634-648.
12. Emerson R.N., Kennady C.J.,Ganesan S., "Effect of Organic additives on the Magnetic properties of Electrodeposition of CoNiP Hard Magnetic Films", Thin solid films, 2007,515, 3391-3396.
13. Hamid Z.A., "Electrodeposition of Cobalt- Tungsten Alloys from Acidic Bath Containing Cationic Surfactants", Materials Letters.,2003,57, 2558.
14. Kannan, R, Ganesan, S,Selvakumari, "Structural and Magnetic properties of electrodeposited NiFeWS thin films", Optoelectronics and advanced materials-Rapid Communication 2012, 3-4, 383-388.
15. Kannan, R, Ganesan, S ,Selvakumari, TM "Synthesis and characterization of nano crystalline NiFeWS thin films in diammonium citrate bath", Digest journal of nanomaterials and biostructures, 2012,7, 1039-1050.
16. Nosang V, Park D.Y, Yoob B.Y. and Paulo T.A., "Development of electroplated magnetic materials for MEMS", Journal of Magnetism and Magnetic Materials., 2003, 265, 189-198
17. Cho H. J, Bhansali S. ,Ahn C. H., "Electroplated thick permanent magnet arrays with controlled direction of magnetization for MEMS application", Journal of Applied Physics, 2000,87, 6340- 6342.
18. Esther. P, Joseph Kennady.C., "Effect of sodium tungstate on the properties of Electrodeposited nanocrystalline Ni-Fe-W films", Journal of Non Oxide Glasses., 2010, 1, 35-44.