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SYNTHESIS AND CHARACTERIZATION OF Fe-Ni-Co MAGNETIC THIN FILMS AT DIFFERENT BATH TEMPERATURE

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ABSTRACT

The magnetic alloy thin films Fe-Ni-Co were deposited on the copper substrateby electrodeposition method at different temperature. Electro deposited Fe-Ni-Co thin films were subjected to the morphological, structural, and mechanical characterization analysis. The chemical composition of the coated films was analysed by EDAX. The surface and structural morphology of the coated film were analysed by using SEM and XRD. The mechanical properties of Fe-Ni-Co thin films have been analysed by VHT. The electroplated Fe-Ni-Co thin films were strongly adherent to the copper substrate. The SEM pictures of Fe-Ni-Co thin films show that, the deposits of thin films are crack free, uniform and bright surface with fine grain size. All the electro deposited Fe-Ni-Co films exhibit FCC crystalline structure. The VHN result of Fe-Ni-Co thin films shows that the Fe-Ni-Co 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

Electrodeposited nickel is one of the most widely used materials in the fabrication of micro machines such as micro cantilevers, micro gears and their components (14,22,26). Electrodeposition is the dominant manufacturing technology in many new applications such as MEMS devices, NEMS devices, data storage media and magnetic recording head. The most commonly used magnetic materials in MEMS and NEMS are soft magnetic materials, such as NiCo ,NiFe and NiP (1-6) .The combination of good mechanical properties and high corrosion resistance has led to the use of electroplated NiFe films in microscopic sensors, actuators and systems (15-18). The use of NiFe

as the soft film can be improved by adding a third element with NiFe alloy. Electro depositedPermalloy[NiFe] is thebestknownthinfilm alloyinMEMS applications(13,19). In this current investigation, the electrodeposition method has been chosen for coating Fe-Ni-Co thin films. In this present work, we have analysedthe effect of different temperatureon FeNiCothin films with. This paper summarizes the synthesis and characterizations of electroplated FeNiCo thin films.

II EXPERIMENTAL PART

The working conditions and bath composition of Fe-Ni-Co alloy thin film are shown in Table 1.The Fe-Ni-Co 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 breath and 7.5cm as length were used as substrates. Both cathode and anode were washed with soap and soaking in 15% H_2SO_4 for 2 minutes. The reagent grade chemicals and triple distilled water were used to prepare electroplating bath . The pH value of the bath was adjusted to 6 by adding few drops of ammonia solution(10-14). The Fe-Ni-Co thin films were electro deposited on the copper substrate by applying a current of 15 mA for 15 minutes at30˚C,50˚C ,70˚C and 90˚C. The cathode was carefully removed from the bath after 15 minutes and dried for few minutes. The surface morphology of the Fe-Ni-Co thin films was analysed with the help of Scanning electron microscope (SEM). The film composition and structural characters of thin films were measured by Energy-dispersive X-ray Spectroscopy (EDAX) andX-ray diffraction (XRD) respectively. The hardness of Fe-Ni-Co thin films was measured by Vickers Hardness Test (VHN).The magnetic property of Fe-Ni-Co thin films film was measured by Vibrating Sample Magnetometer (VSM).The thicknesses of the films were determined by cross sectional view of SEM images. The electrodeposition bath details of Fe-Ni-Co thin films are given in table 1.

Table 1. Electroplating bath details of FeNiCo thin films

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

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III RESULTS AND DISCUSSION

3.1 Composition of Electrodeposited Thin Films

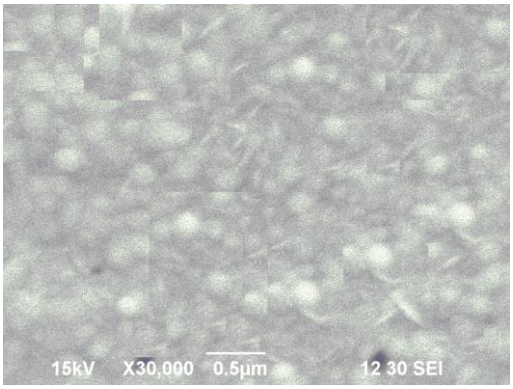
The chemical composition of the electroplated thin films is analysed by EDAX spectrum. The EDAX data's of thin films are shown in Table 2.EDAX result showed that the films obtained at higher temperature have high ferrous content. The highest ferrous content of 22.13 wt% was obtained at temperature 90˚C.EDAX result showed that Ni content increases with increasing the bath temperature. The maximum Ni content of 47.92 wt% was obtained for Fe-Ni-Co thin films at 90˚C bath temperature. The weight percentage of Co decreaseswhile increasing the bath temperature.Ammonia solution is used to correct the pH value of the bath solution only and its effect on the film was ignored.

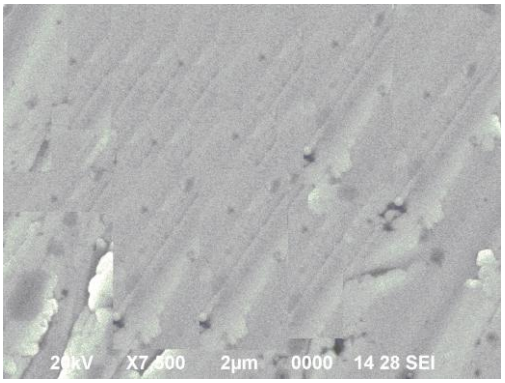
Table 2: EDAX analysis of thin films

S. No	Temperature	Co Wt%	Ni Wt%	Fe Wt%
1.	30˚C	68.45	19.87	11.68
2	50˚C	56.16	29.56	14.28
3	70˚C	40.23	40.08	19.69
4	90˚C	29.95	47.92	22.13

3.2 Morphological Observation

The surface morphology of the electroplated Fe-Ni-Cothin films with different temperature is analysed by using 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 we conclude that the formation of thin films on the copper substrate is uniform in nature





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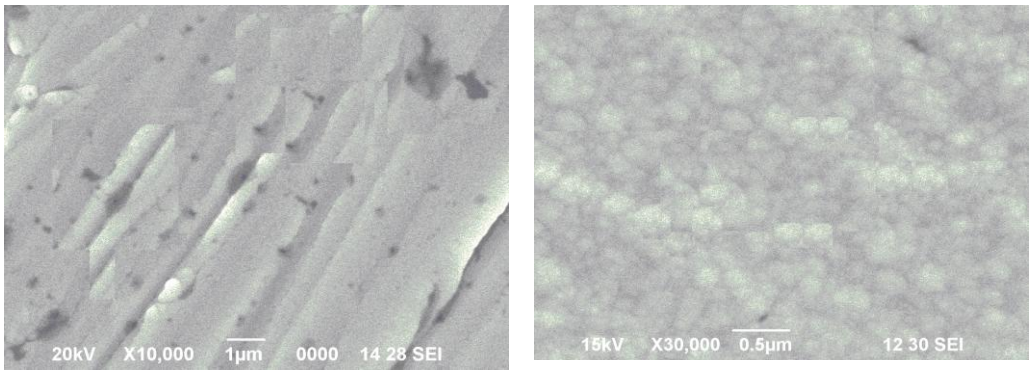


Figure 1: SEM images for Electro deposited Fe-Ni-Cothin film for different bath temperatures

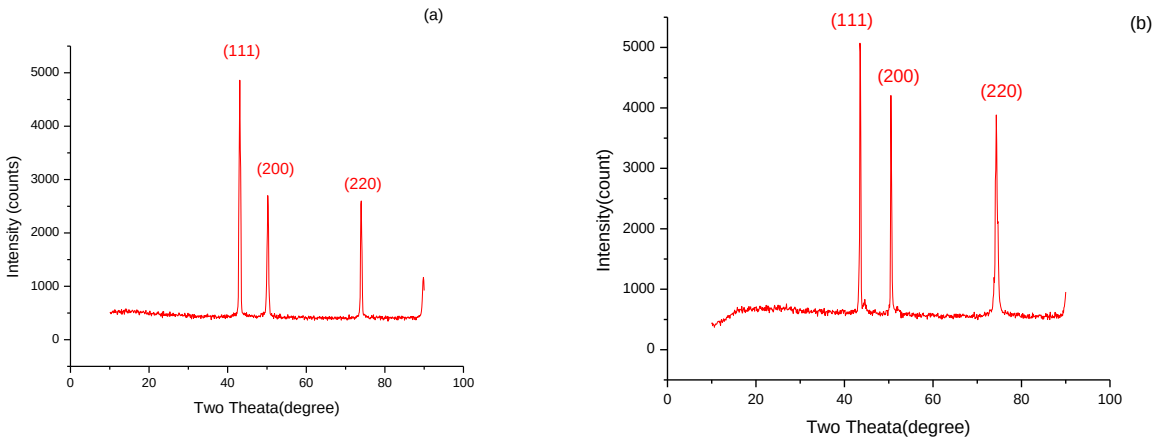
(a) 30˚C (b) 50˚C (c) 70˚C (d) 90˚C

3.3 Structural Analysis

The crystal structure of the electro deposited Fe-Ni-Co alloy thin films was determined by XRD analysis. X- ray diffraction patterns of Fe-Ni-Co films obtained at different temperatures 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 was calculated from XRD usingScherrer'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 and.The XRD patterns of NiCoFe filmsrevealedtheexistence ofFCCphasewith (111), (200) and (220) diffraction peaks.The result shows that the crystalline sizes of the Fe-Ni-Co deposits obtained by electro deposition process are in the nano scale and the average crystallite size was around 21 nm.



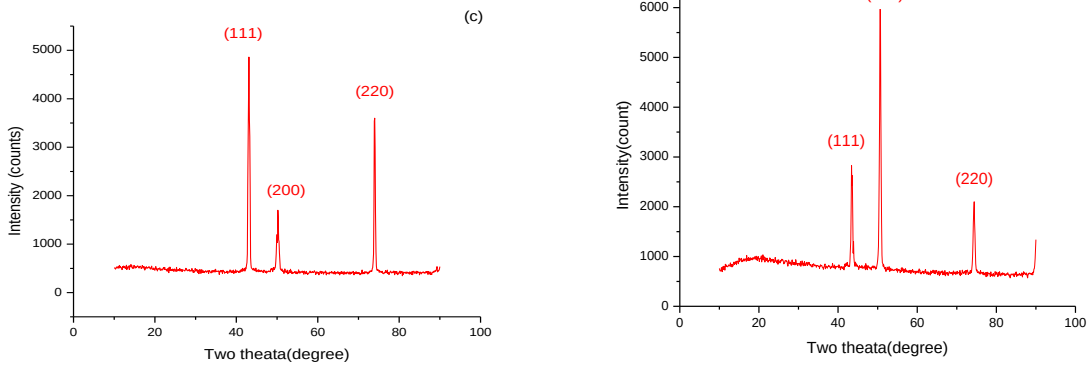


Fig.2 XRD patterns of Fe-Ni-Co thin films at (a) 30^0C (b) 50^0C (c) 70^0C (d) 90^0C

The crystal size of Fe-Ni-Co alloy films is tabulated and shown in table 3.When the bath temperature is increased the crystalline size of thin films decrease due to onset orientation of crystals during electrodeposition

Table.3 :Structural characteristics of NiCoFe alloy thin films

S. No	Bath Temperature (^{0}C)	2Ɵ (deg)	d (A^0)	Particle size, D (nm)	Strain (10^{-3})	Dislocation density (10^{14} / m^2)
1	30	43.310	1.5634	23.32	1.578	21.87
2	50	43.681	1.4376	21.23	1.765	22.43
3	70	43.108	1.7650	19.67	1.854	23.76
4	90	50.412	1.4538	18.02	1.897	26.34

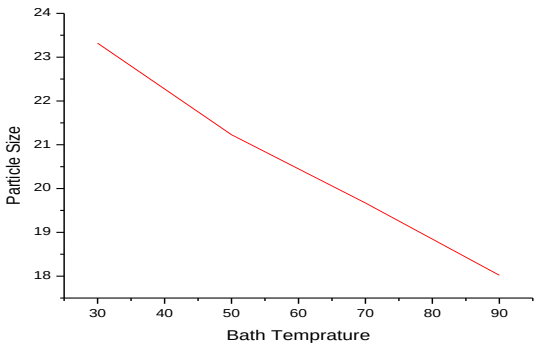


Figure 3. Crystalline Size as a function of Bath temperature

3.4 Mechanical Properties

Hardness of the films was examined by using Vickers hardness tester (the diamond intender method). The results show that the hardness increases with increasing bath temperature.This may be due to lower stress associated with electrodeposited Fe-Ni-Co films. The hardness of Fe-Ni-Co thin films have been shown in table 4.

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

S.No	Bath Temperature (˚C)	Vickers Hardness (VHN)
1	30	76
2	50	87
3	70	102
4	90	106

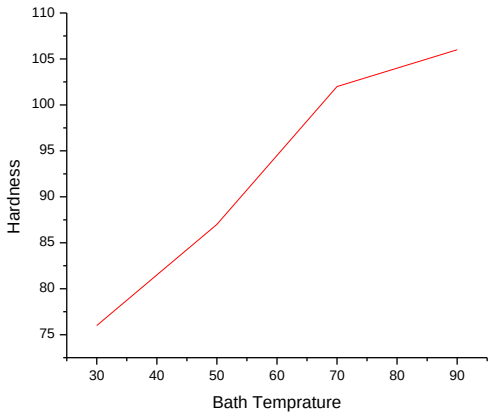


Figure 4. Vickers Hardness as a function of bath temperature

IV CONCLUSION

The Ni-Co-Fe magnetic thin films were successfully prepared by electro deposition at different bath temperatures 30˚C, 50˚, 70˚C and 90˚C .The crystalline sizes of the deposits obtained by electro deposition process are in the nano scale. The thin films obtained at different temperature are uniform, bright andcrack free. FCC was the dominant structure of electro deposited Fe-Ni-Cothin films(14,22). Hardness is increases with increasing bath temperature. When the bath temperature was increasedfrom 30 ˚C to 90 ˚C, the particle size values decreases from 23.32 nm to 18.02 nm. This is 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, MEMS and NEMS.

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