SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF ELECTRODEPOSITED CONIP ALLOY THIN FILM

T. Baskar[1], K.S.Rajni[2], R.Kannan[3]

[1]*Research Scholar, Department Of Physics, Karpagam University, Coimbatore, Tamilnadu, (India)*

[2]*Department of Sciences-Physics, Amrita Vishwa Vidyapeetham, Coimbatore, Tamilnadu, (India)*

[3]*Department of Physics, K.S.R.College of Engineering, Tiruchengode, Tamil Nadu, (India)*

ABSTRACT

The nano crystalline CoNiP thin films were synthesized by electrodeposition method. The CoNiP thin films have been coated on the copper substrate at 30 ˚ C by applying a constant current density of 1 mA/cm^2 for 15 minutes. The electrodeposited CoNiP thin films were subjected to various structural characterization techniques like EDAX, XRD, SEM and Vickers hardness test. EDAX investigation gives the chemical composition of the coated films. The coated CoNiP thin films exhibit the maximum Co content of 59.87 wt%, Ni content of 26.48 wt% and 13.66 wt% of P. The Surface morphology of the coated film has been analyzed by using SEM micrographs. The electroplated CoNiP thin films are smooth, uniform and adherent with substrate. X-ray diffraction analysis of CoNiP thin films reveals the existence of nano crystalline FCC phase with three predominant diffraction peaks. The average crystalline size of CoNiP thin films were calculated from XRD is in range of 32 nm. The Vickers hardness test shows 41 VHN while applying the load of 25g. The electroplated CoNiP thin films were strongly adherent to the substrate. This was observed from bend and scratch test. Generally CoNi based thin films have potential applications in the fabrication of MEMS and NEMS devices. Based on the magnetic properties of CoNiP thin films may be used in MEMS devices.

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

I. INTRODUCTION

In power electronics, for the production of super capacitors now a days CoNi oxide thin films are the suitable materials. The Fe, Ni, Co, W, Cr based thin films have potential applications in the field of magnetic sensor technology, computer read/write heads micro electro mechanical systems (MEMS), Nano electro mechanical systems (NEMS) and large scale integration (ULSI) devices based on their compositions, structural properties and magnetic properties. In the current MEMS technologies, CoNi, CoW, NiW and NiFe thin film alloys are used because those films can exhibit excellent magnetic properties [1-2].

The CoNiP alloy thin films can be synthesized through several physical and chemical methods such as thermal decomposition method, co-precipitation method, spray pyrolysis and electrodeposition method. In this current investigation, the electrodeposition method have been chosen for coating the CoNiP thin films. Because compared to other sophisticated methods electrodeposition has several advantages. The electrodeposition

method is the simplest, most economical, reliable and reproducible technique. Electrodeposition of CoNi films and their studies were carried out by few numbers of researchers [15-26].

Normally CoNi thin films have good magnetic properties so that it can be used in various applications. If we add third element to CoNi the structural, mechanical and magnetic properties may be enhanced. In this present work we planned to analyze the effect of P on CoNi thin films in order to exploit the full potential of CoNiP thin films. This paper summarizes the synthesis and structural characterizations of electroplated CoNiP thin films.

II. EXPERIMENTAL PART

The CoNiP 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. The electroplated bath details of CoNiP thin films are shown in Table 1. 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 15% 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 CoNiP 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 CoNiP 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 CoNiP thin films is shown in fig 1.

Table 1: Electrode Position Bath Details of CoNiP Thin Films

S. No	Name of the chemicals and parameters	Data g/1
1.	Nickel Sulphate	30
2.	Cobalt Sulphate	15
3.	Phosphorous acid	10
4.	Ammonium citrate	40
5.	Citric acid	8.5
6.	Boric acid	10
7.	pH value	6
8.	Time Duration	15 min
9.	Current density	1 mA/cm^2

Figure 1: Experimental setup of electrodeposited CoNiP thin films

III. RESULT AND DISCUSSION

3.1 Composition of the Electro Deposited Conip Thin Films

The chemical composition of the electroplated CoNiP thin films is analyzed by EDAX spectrum. The EDAX spectrum of CoNiP thin films are shown in fig 2. The EDAX data of CoNiP thin films are shown in Table 2.

Table 2: EDAX analysis of CoNiP thin films

S. No	Temperature	Co Wt%	Ni Wt%	P Wt%
1.	Room Temperature	59.87	26.48	13.66

From EDAX, we conclude that, the electroplated thin films have maximum Co content of 59.87 wt% , 26.48 wt% of Ni and 13.66 wt% of P.

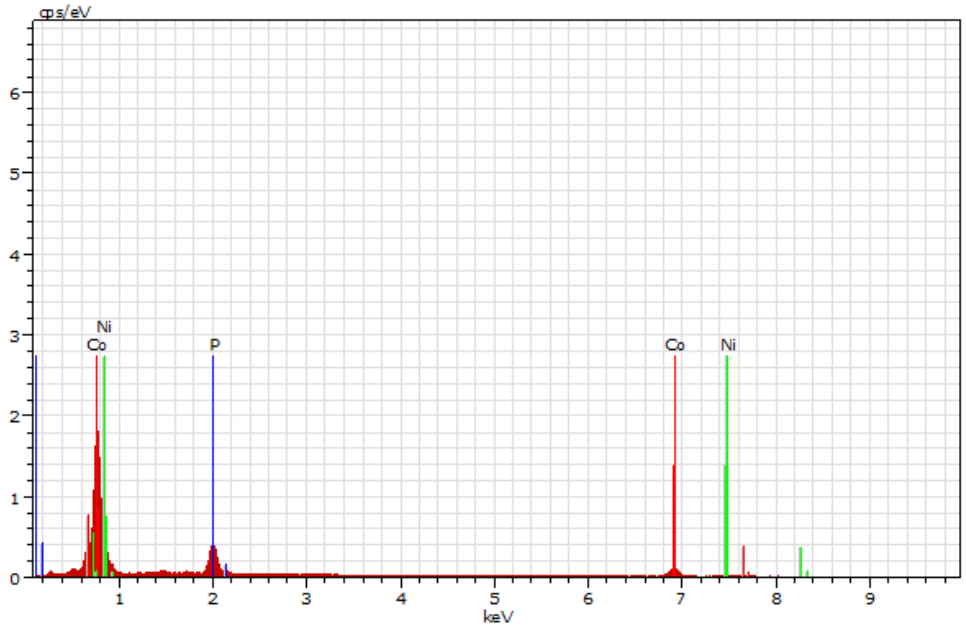

Figure 2: EDAX spectrum of CoNiP thin film

3.2 Surface Morphology of CoNiP Thin Films

The surface morphology of the electroplated CoNiP thin films is analyzed by using SEM pictures and are shown in fig 3.The electroplated CoNiP thin films are smooth, uniform and adherent with substrate. The CoNiP thin films are crack free, bright and uniform. From SEM analysis we conclude that the formation of thin films on the copper substrate is uniform in nature. The thickness of the coated CoNiP thin films is 2.37 µm determined from cross sectional view of SEM images.

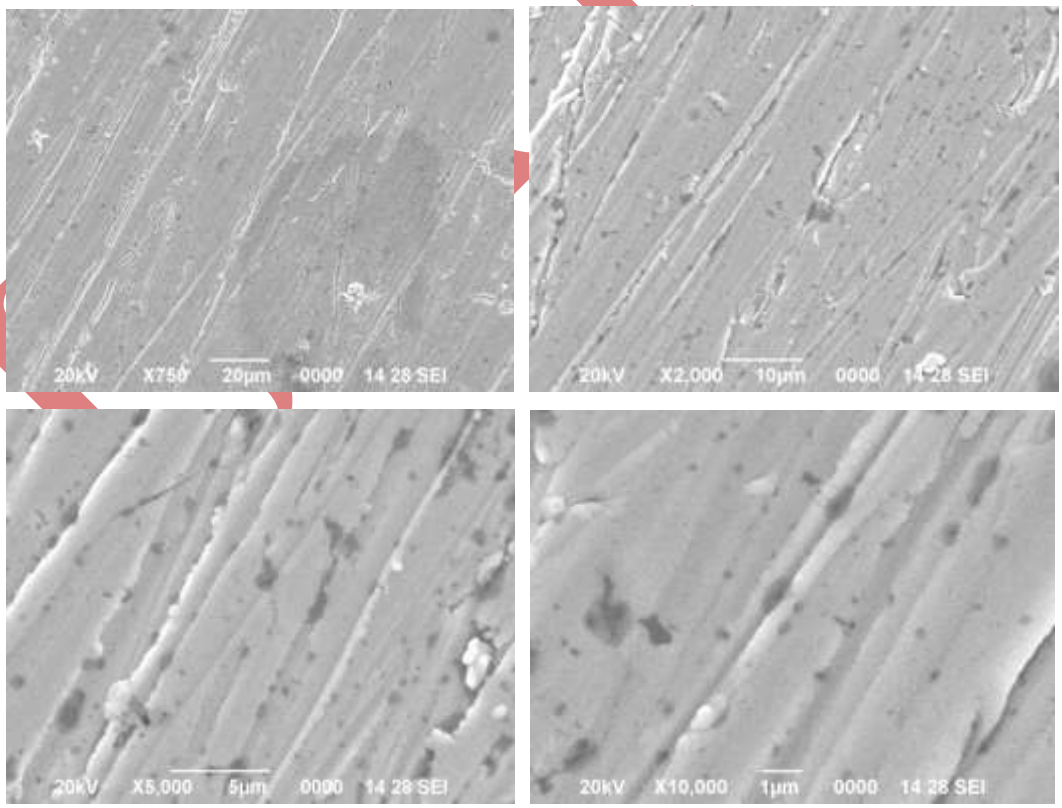

Figure 3: SEM Images of Electrodeposited CoNiP Thin Films

3.3 Structural Properties Of CoNiP Thim Films

The crystal structure of the electro deposited CoNiP alloy thin films was determined by XRD analysis. X- ray diffraction patterns of CoNiP 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 were calculated from XRD pattern using formula

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

The data's obtained from XRD analysis are compared with standard JCPDS data and found to have FCC crystalline structure with three predominant peaks of (200), (111) and (220). These values clearly show that the crystalline sizes of the CoNiP deposits obtained by electro deposition process are in the nano scale. The crystal size of CoNiP alloy films are tabulated as shown in Table 3. The average crystalline size of the CoNiP thin films are around 32 nm.

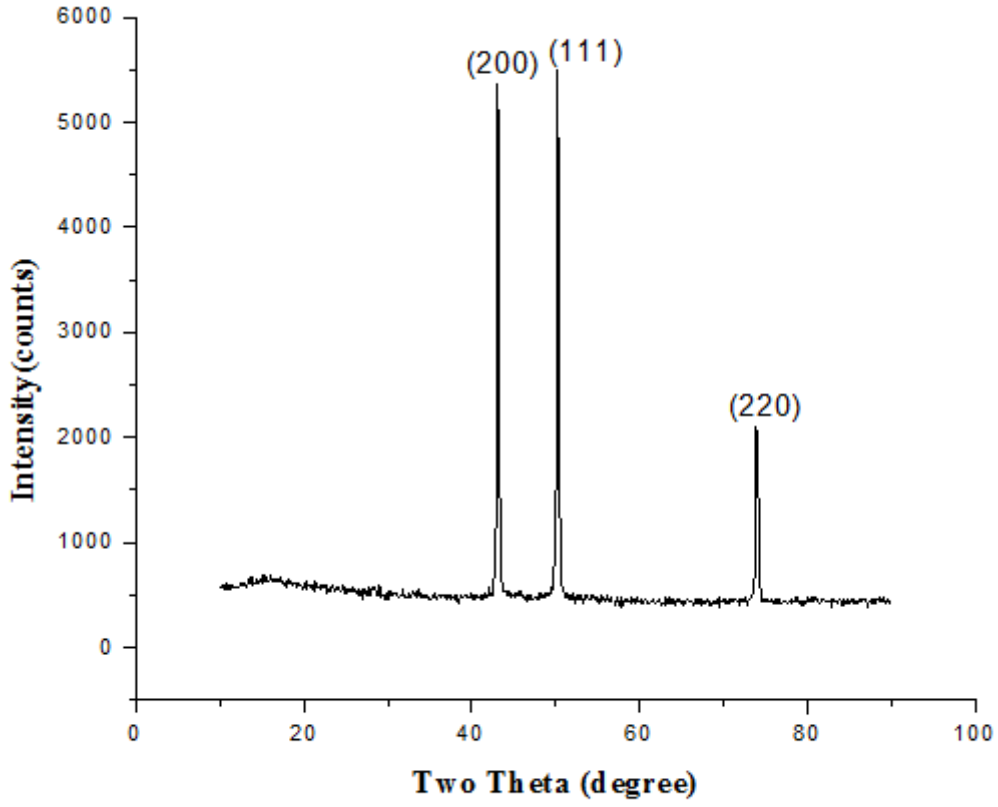

Figure 4: XRD Pattern of Electrodeposited CoNiP Thin Film

From XRD we conclude that the CoNiP 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 CoNiP thin films may be decreased due to onset orientation of crystals during electrodeposition. So we planned to investigate the magnetic properties of CoNiP thin films with different bath temperature.

Table 3: Crystalline size of CoNiP alloy thin films

S.No	2θ (deg)	d value (A°)	Crystalline size D nm	Strain 10^{-4}	Dislocation density ($10^{14}/m^2$)
1	43.08	2.0977	31.74	11.575	9.9267
2	50.2	1.8157	30.40	12.085	10.8206
3	73.93	1.2809	34.91	10.525	8.2084

3.4 Mechanical Properties

Adhesion of the electrodeposited CoNiP thin film with the substrate is tested by bend test. 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 CoNiP 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 41 VHN while applying the Load of 25g.

IV. CONCLUSION

The CoNiP 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 CoNiP thin films. The crystalline sizes of the CoNiP deposits obtained by electro deposition process are in the nano scale. The average crystalline size of CoNiP films is around 32 nm. Hardness of this magnetic thin film is 41 VHN. This article summaries the optimized operating condition of CoNiP electroplated bath. Based on their magnetic properties of CoNiP thin films, these films may be used in various electronic devices including high density recording media, magnetic actuators, magnetic shielding, magnetic writing heads, high performance transformer cores and MEMS. Because of the potential applications of CoNi based thin films in various industrial areas we planned to analyze the magnetic property of CoNiP 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] Nosang V.Myung, D.-Y Park, B.-Y.Yoo, Paulo T.A.Sumodjo, J. of Magn and Mag Mat, **265,** 189-198(2003).

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

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

[6] T.M.Selvakumari, P.Muthukumar, S.Ganesan, Digest J. of Nanomaterials and Biostructures, **5,** No4, p.903-907(2010).

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

[8] A. N. Correia and S.A.S.Machado, Electrodeposition and Characterisation of Thin layers of Ni-Co Alloys obtained from Dilute Chloride Baths, *Electrochimica Acta*, **Vol.45, pp.1733-1740,(2000),**

[9] Bai and C. –C. Hu, "Iron-Cobalt and Iron-Cobalt-Nickel Nanowires deposited by Means of Cyclic Voltammetry and Pulse-Reverse Electroplating". *ElectrochemistryCommuincations, Vol.5, pp.78-82***(2003).**

[10] D. Golodnitshy, Y. Rosenberg and A.Ulus, "The Role of Anion Additives in the Electrodeposition of Nickel-/Cobalt Alloys from *Sulfamate Electrolyte," ElectrochimicaActa, Vol.47, pp.2707-2714*, **(2002).**

[11] A. Bai and C. –C. Hu, "Composition controlling of Co-Ni and Fe-c=Co Alloys Using Pluse-Reverse Electroplating through means of Experimental Strategies," *ElectrochimicaActa, Vol.50, pp. 1335-1345***(2005).**

[12] B. Chi, J. Li, K. Yang, Y. Gong and N. Wang, "Depositon of Ni-Co by Cyclic Voltammetry method and its Electrocatalytic properties for oxygen Evolution reaction, " International journal of hydreogen Energy, Vol.30, **(2009).**

[13] E. Gomez, S.Pane and E.Valles, "Electrodeposition of Co-Ni and Co-Ni-Cu Systems in Sulpate- Citrate medium, *ElectrochimicaActa, Vol.50, pp. 146-153***(2005).**

[14] Sunil Ganpat Kandalkar, Hae-Min Lee, Seung Hey Seo, Kangtaek Lee and Chang-Koo Kim, Preparation and Characterization of Electrodeposited NiCo Oxide thin films for electrochemical capacitors, *Korean J. Chem. Eng*, 28(6), 2011, 1461-1467.