SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF ELECTRO DEPOSITED NICKEL COBALT BORON ALLOY THIN FILM

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ABSTRACT

Nano crystalline Ni-Co-B alloy thin films were deposited by electrodeposition method on the copper substrate at 32˚C .The structural, chemical composition, surface morphology and mechanical property of electro deposited Ni-Co-B thin films were studied. EDAX investigation gives the chemical composition of the coated films. The structural and surface morphology of the coated film have been analyzed by using XRD and SEM micrographs respectively. The mechanical properties of coated films were studied by Vickers hardness test. The electroplated thin films were strongly adherent to the substrate. This was observed from bend and scratch test. The deposits of thin films were found to be smooth and nano crystalline. The coated Ni-CO-B thin films exhibit the maximum Co content of 65.10 wt%, Ni content of 27.15 wt% and 7.75 wt% of B. The Vickers hardness test shows 44 VHN while applying the load of 25g .Generally CoNi based thin films have potential applications in the fabrication of MEMS and NEMS devices. Based on the magnetic properties of thin films may be used in MEMS devices. The average crystalline size of Co-Ni-B thin films were calculated from XRD is in range of 29.93 nm

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

I INTRODUCTION

Now a day's CoNi oxide thin films are the suitable materials for the production of super capacitors in power electronics industry. The Ni, Co, W, Fe,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 properties and magnetic properties.

In the current MEMS technologies, CoNi, CoW, NiFe 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 have been chosen for coating the Ni-Co-B thin films. Because compared to other coating 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 [15-20]. 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 B on CoNi thin films. This paper summarizes the synthesis and structural characterizations of electroplated thin films. Very few research works are documented about the structural and composition of electrodeposited crystalline Ni-Co-B alloys .

II EXPERIMENTAL PART

The bath composition of Ni-Co-B 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-Co-B 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-Co-B 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-Co-B thin films is shown in fig 1.

Table 1: Electro deposition Bath Details of Thin Films

S. No	Name of the chemicals and parameters	Data g/1
1.	Nickel Sulphate	30
2.	Cobalt Sulphate	15
3.	Borax	10
4.	Sodium chloride	40
5.	Saccharin	5
6.	Boric acid	20
7.	pH value	6
8.	Time Duration	15 min
9.	Current density	1 mA/cm^2

Figure 1: Experimental setup of electrodeposited Ni-Co-B thin films

III RESULT AND DISCUSSION

3.1 Composition of the Electro Deposited Thin Films

The chemical composition of the electroplated thin films is analyzed by EDAX spectrum. The EDAX spectrum of Ni-Co-B 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	Co Wt%	Ni Wt%	B Wt%
1.	Room Temperature	65.10	27.15	7.75

From EDAX, we conclude that, the electroplated thin films have maximum Co content of 65.10 wt% , 27.15 wt% of Ni and 7.75wt% of B.

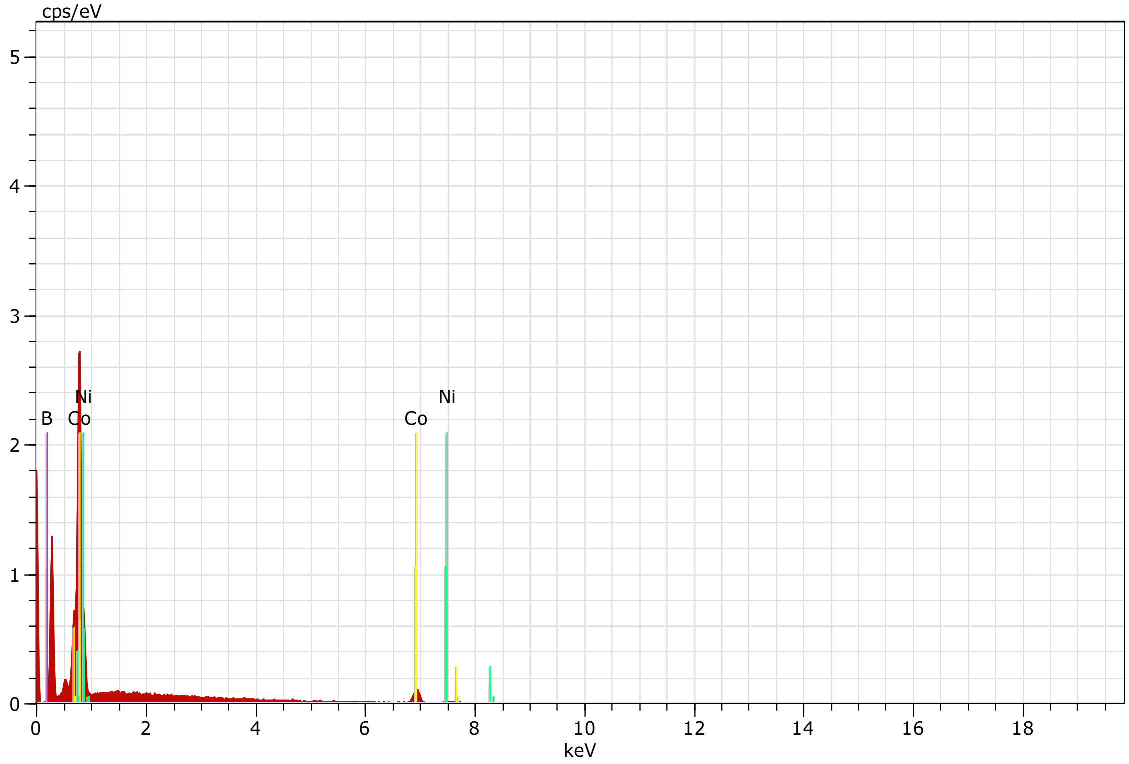

Figure 2: EDAX spectrum of thin film

3.2 Surface Morphology of Thin Films

The surface morphology of the electroplated Ni-Co-B 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, 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 thin films is 2.27 µm determined from cross sectional view of SEM images.

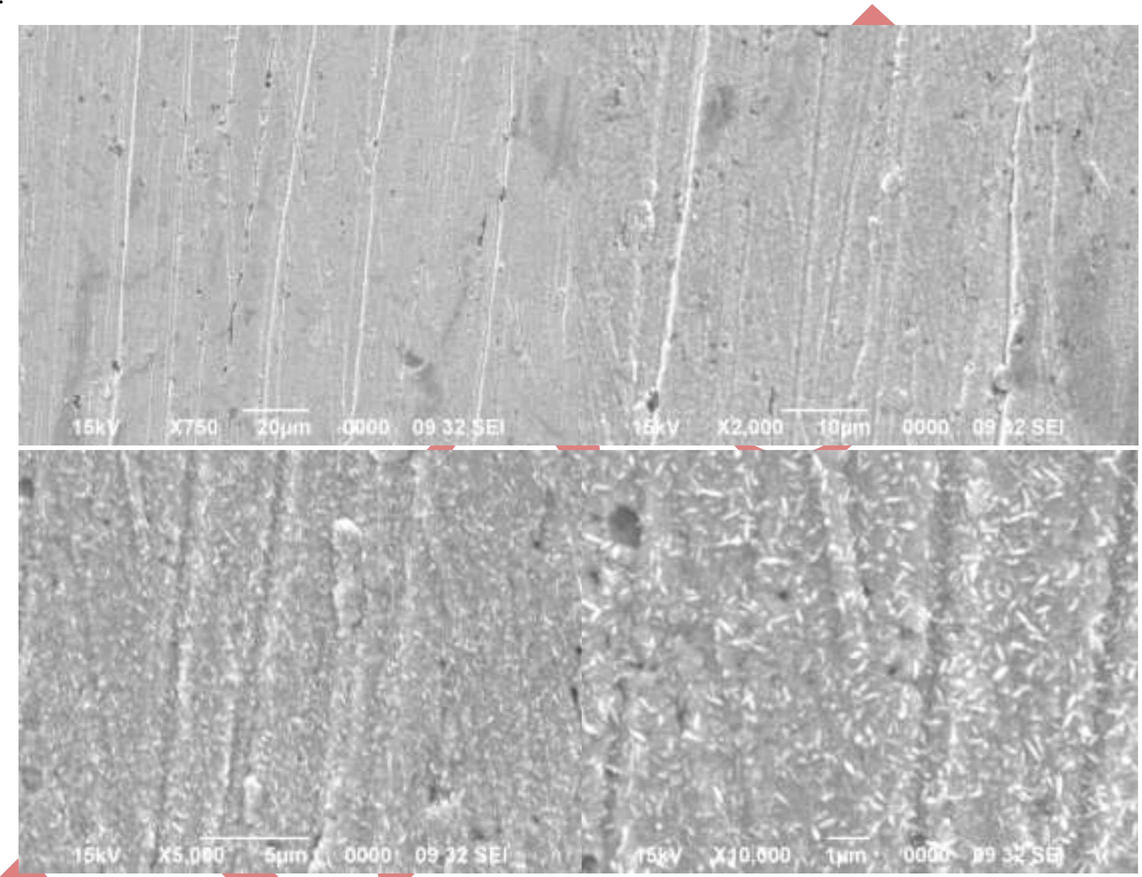

Figure 3: SEM images of electrodeposited thin films

3.3 Structural Properties of Ni-Co-B Thim Films

The crystal structure of the electro deposited Ni-Co-B 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-Co-B thin films were calculated from XRD pattern using formula

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

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) , (220) and (311). 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 29.93 nm.

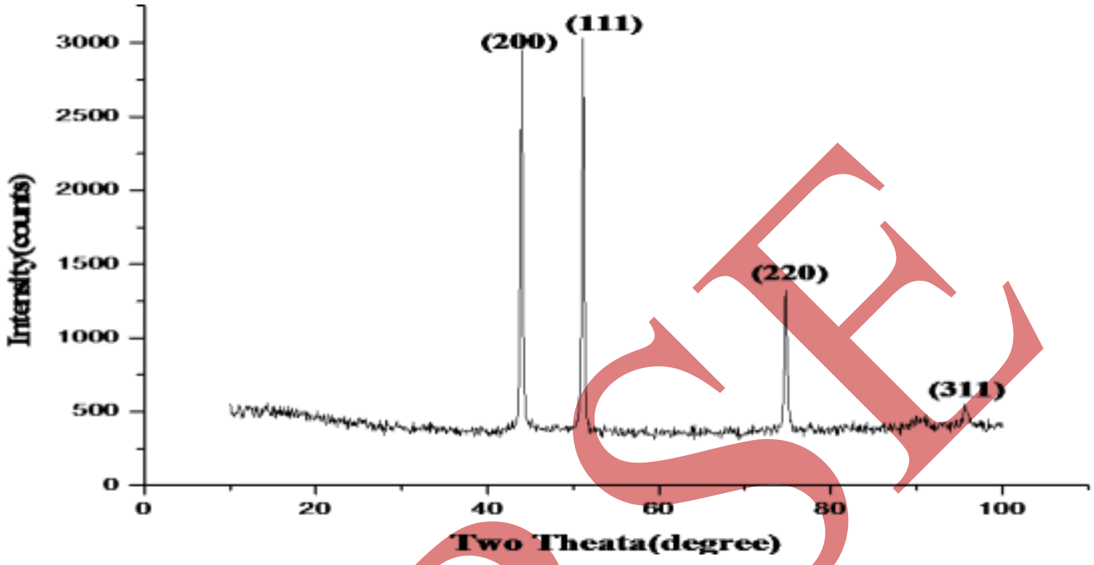

Figure 4: XRD pattern of electrodeposited thin film.

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 thin films may be decreased due to onset orientation of crystals during electrodeposition. So we planned to investigate the magnetic properties of thin films with different bath temperature and different boron concentration.

Table 3: Crystalline size of alloy thin films

S.No	**2θ (deg)**	**d value (A˚)**	**Crystalline size D Nm**	**Strain 10^{-4}**	**Dislocation density $(10^{14}/m^2)$**
1	43.94	2.0588	29.08	12.6319	11.8191
2	51.07	1.7869	26.85	13.6863	13.8745
3	74.68	1.2700	29.85	12.3080	11.2208
4.	95.61	1.0398	33.97	10.8174	8.6675

3.4 Mechanical Properties

Adhesion of the electrodeposited 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 Ni-Co-B 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 44 VHN while applying the Load of 25g.

IV CONCLUSION

The Ni-Co-B 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-Co-B thin films. The crystalline sizes of the deposits obtained by electro deposition process are in the nano scale. The average crystalline size of Ni-Co-B films is around 29.93 nm. Hardness of this magnetic thin film is 44 VHN. This article summaries the optimized operating condition of electroplated bath. Based on their magnetic properties of 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 thin films in our future research work.

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