International Journal of Thin Films Science and Technology

http://dx.doi.org/10.12785/ijtfst/040111

Synthesis and Structural Characterization of CoNiW alloy Thin Films by Electrodeposition.

R. Kannan[1,*] *and S. Kokila*[2].

[1]Department of Physics, KSR College of Engineering, Tiruchengode-637215, Tamil Nadu, India.
[2]Department of Physics, K.S. Rangasamy College of Arts and Science, Tiruchengode-637215, Tamil Nadu, India.

Received: 6 Aug. 2014, Revised: 18 Nov. 2014, Accepted: 20 Nov. 2014.
Published online: 1 Jan. 2015.

Abstract: Nanocrystalline CoNiW thin films were synthesized by electrodeposition method. The CoNiW thin films have been coated on the copper substrate by applying a constant current of 7.5 mA for 15 minutes. The CoNiW coated thin films were subjected to various characterization techniques like EDAX, XRD, SEM and Vickers hardness test. EDAX investigation gives the chemical composition of the coated films. The Co content of 75.37 wt%, Ni content of 13.67 wt% and 10.96 wt% of W were determined from EDAX analysis. The surface morphology of the coated film is analyzed by using SEM photographs. X- ray diffraction showed that the existence of nanocrystalline phase of CoNiW. The average crystalline size of CoNiW thin films were calculated from XRD is in the range of 34 nm. The Vickers hardness of 105 VHN while applying the load of 25 g. The electroplated CoNiW thin films were strongly adherent to the substrate. This was observed from bend and scratch test.

Keywords: CoNiW thin films, electrodeposition, characterization techniques, crystalline size, surface morphology and adhesion.

1.Introduction

The elements like Fe, Ni, Co, W, Cr have potential applications based on their compositions and structural properties 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. In the current MEMS technologies may use CoNi, CoW, NiW, NiFe are the suitable alloys because of their excellent magnetic properties [1- 2]. In this research work the CoNiW films are coated by electrodeposition method. Compared to other physical chemical methods, electrodeposition method have several advantages like low manufacturing cost, easy and simple method, easy to oprate etc., [3-11]. NiCo alloys are the suitable magnetic materials which can be used in several fields such as MEMS and NEMS. The addition of W to NiCo alloys may enhance their structural, mechanical and magnetic properties. The effect of W on CoNi alloys may give good corrosion resistance, ductility, good mechanical strength, hardness, and magnetic properties [12-14]. Electrodeposition of CoNi films and their studies were carried out by a number of researchers [15-26]. So we planned to investigate the effect of W on CoNi alloy thin films coated from tri sodium citrate bath in order to exploit the full potentials of CoNiW films. This paper summarizes the synthesis and structural characterizations of electroplated CoNiW thin films.

2. Experimental section

A copper plate of size 1.5cm as breath and 7cm as length were used for substrates. Copper substrate act as the cathode and pure stainless act as the anode. Both cathode and anode were pre-treated by washing with soap and soaking in 10% H_2SO_4 for 2 minutes. Just before the deposition both the plates are degreased by acetone.

The electroplating bath prepared by all the reagent grade chemicals is dissolved in triple distilled water. CoNiW thin films were electrodeposited on the copper substrate using relevant salts in tri-sodium citrate bath at room temperature. The chemical composition and operating conditions of the electroplating bath are as shown in Table 1. An adhesive tape was used to mask off all the substrate except the area on which the deposition of films was desired. All the reagent grade chemicals were dissolved in triply distilled water. The film was deposited on copper substrate by applying a constant current of 7.5 mA for a period of 15 minutes at room temperature

Table 1: List of chemical composition and operating conditions.

S. No	Name of the chemicals and parameters	Data g/l
1	Nickel sulphate	30
2	Cobalt sulphate	15
3	Sodium tungstate	7.5
4	tri sodium 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

*Corresponding author e-mail: kannanarjun13@gmail.com

3. Result and Discussion

3.1. Composition of the electro deposited CoNiW thin films.

The chemical composition of the electroplated thin films are analysed by EDAX-spectrum. The EDAX spectrum of CoNiW thin films are shown in Figure 14. The EDAX data of CoNiW thin films are shown in Table 2.

Table 2: EDAX analysis of CoNiW thin films.

S.No	Temperature	Co Wt%	Ni Wt%	W Wt%
1	Room Temperature	75.37	13.67	10.96

Fig.1: Experimental set up of electrodeposited NiCoW thin films.

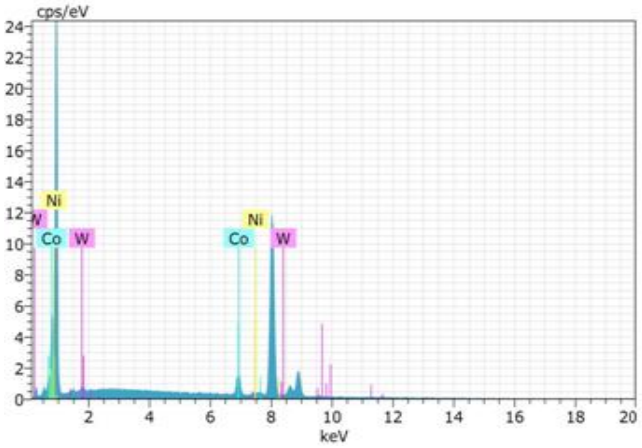

Fig.2: EDAX spectrum of Co-Ni-W thin film

From EDAX, we conclude that, the electroplated thin film have 75.37 at% of Co, 13.67 at% of Ni and 10.96 at % of W.

3.2. Surface morphology of CoNiW thin films

The surface morphology of the electroplated CoNiW thin films are analyzed by using SEM pictures. The SEM pictures of CoNiW thin films are shown in Figure 3. The electroplated CoNiW thin films are smooth, uniform and adherent with substrate and gray in appearance. The existence of micro voids and cracks are due to internal stress. From SEM analysis we conclude that, the formation of thin films on the copper substrate is uniform in nature.

3.3. Structural properties of CoNiW thin films

The crystal structure of the electro deposited CoNiW alloy thin films was determined by XRD analysis. X-ray diffraction patterns of CoNiW films obtained from tri-sodium citrate bath at room temperature were shown in Figure 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 the XRD pattern using the formula

$$(D=0.954\lambda/\beta\cos\theta) \quad (1)$$

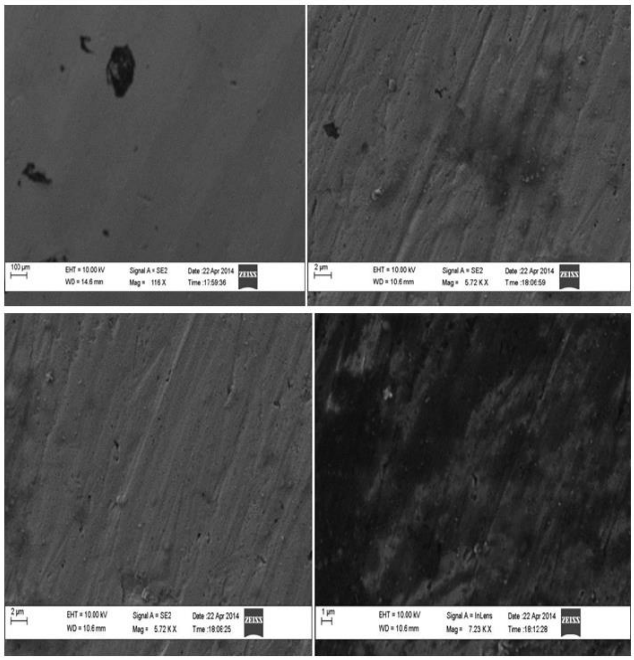

Fig.3: SEM images of electrodeposited Co-Ni-W thin films.

These values clearly show that the crystalline sizes of the CoNiW deposits obtained by electro deposition process are in the nanoscale. The crystal size of CoNiW alloy films obtained from tri-sodium citrate bath are tabulated as shown in Table 3. The XRD patterns of CoNiW films revealed the existence of FCC phase with (111), (130) and (420) diffraction peaks. The crystalline size decreases with increase in bath concentration.

Table 3: Crystalline size of Co-Ni-W alloy thin films.

2 θ (deg)	Lattice Parameter (A^0)	Crystalline size D nm	Strain 10^{-4}	Dislocation density (10^{14} / m^2)
42.93	2.10	34.31	8.90	8.4946

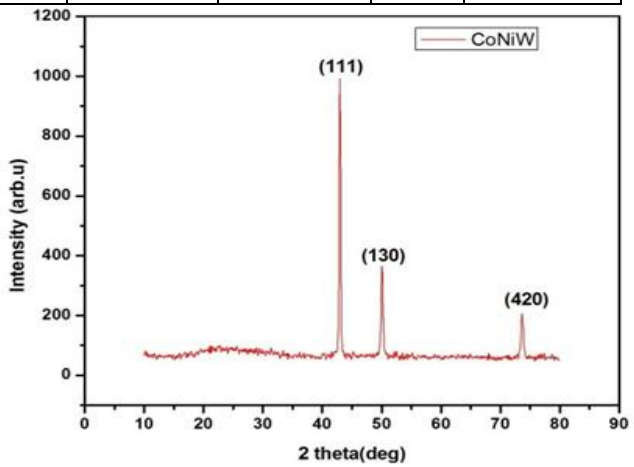

Fig.4: XRD pattern of electrodeposited Co-Ni-W thin film

3.4 Mechanical Properties

Adhesion of the film with the substrate is tested by bend (bending the film with substrate to 180 ˚) test and scratch test. Draw equal lines by pin and paste an adhesive tape over the scratch and pull it. 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 using a Vickers hardness tester by the diamond intender method. Vickers hardness test value is low in the order of 105 VHT while applying the load of 25g.

4. Conclusion

CoNiW magnetic thin films were synthesized by electro deposition from tri-sodium Citrate bath concentration at room temperature. The nano crystalline films obtained at room temperature from higher concentration of citrate bath are crack free and uniform. FCC was the dominant structure of electro deposited CoNiW thin films. The crystalline sizes of the CoNiW deposits obtained by electro deposition process are in the nano scale. The average crystalline size of CoNiW films is around 34 nm. Hardness of this magnetic thin film is 105 VHN. Basedon their structural, mechanical and magnetic properties, these films can be used in various electronic devices including high density recording media, magnetic actuators, magnetic shielding, and magnetic writing heads high performance transformer cores and MEMS.

References

[1] S. D. Leith and D. T. Schwartz, *J. Microelectromech. Syst.* **8**, 384 (1999).

[2] P. Shao, Z. Rummler, and W. K. Schomburg, *J. Micromech. Microeng.* **14**, 305 (2004).

[3] J. Gobet, F. Cardot, J. Bergqvist, and F. Rudolf, *J. Micromech. Microeng.* **3**, 123 (1993).

[4] W. H. Teh, J. K. Luo, M. A. Graham, A. Pavlov, and C. G. Smith, *J. Micromech. Microeng.* **13**, 591 (2003).

[5] S. Majumder, N. E. McGruer, and P. Zavracky, *J. Vac. Sci. Technol. A*, **15**, 1246 (1997).

[6] K. Kataoka, S. Kawamura, T. Itoh, K. Ishikawa, H. Honma, and T. Suga, *Sens. Actuators, A*, **A103**, 116 (2003).

[7] T. Matsunaga, K. Kondoh, M. Kumagae, H. Kawata, M. Yasuda, K. Murata, and M. Yoshitake, *Jpn. J. Appl. Phys., Part 1*, **39**, 7115, (2000).

[8] J. A. Wright and Y. C. Tai, in *Proceedings of the 46th Annual International Relay Conference–NARM'98*, Oak Brook, Illinois, 13-1, April (1998).

[9] J. W. Judy, R. S. Muller, and H. H. Zappe, *J. Microelectromech. Syst.* **4**, 162 (1995).

[10] S. Kawahito, Y. Sasaki, M. Ishida, and T. Nakamura, Digest Tech. Papers, Transducers 93' 888, (1993).

[11] W. Daniau, S. Ballandras, L. Kubat, J. Hardin, G. Martin, and S. Basrour, *J. Micromech. Microeng.*,**5**, 270, (1995)

[12] C. A. Moina and M. Vazdar, Electrodeposition of Nano- Sized Nuclei of Magnetic Co-Ni Alloys onto n-Si (100),*Electrochemistry Communications*, Vol. **3**, pp. 159- 163, (2001).

[13] M. Srivastava, V. E. Selvi, V. K. W. Grips and K. S. Rajam, Corrosion Resistance and Microstructure of Electrodeposited Nickel–Cobalt Alloy Coatings, *Surface &Coatings Technology* **201,** pp. 3051-3060, (2006).

[14] L. Peter, J. Padar, E. Toth-Kadar, A. Cziraki, P. Soki, L. Pogany and I. Bakonyi, Electrodeposition of Co–Ni–Cu/ Cu Multilayers 1. Composition, Structure and Magnetotransport Properties, *Electrochimica Acta*, Vol. **52**, (2007).

[15] 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).

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

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

[18] A. Bai and C.-C. Hu, "Composition Controlling of Co–Ni and Fe–Co Alloys Using Pulse-Reverse Electroplating through Means of Experimental Strategies," *Electrochimica Acta*, Vol. **50**, pp. 1335-1345, (2005).

[19] E. Gomez, S. Pane and E. Valles, "Electrodeposition of Co–Ni and Co–Ni–Cu Systems in Sulphate–Citrate Medium,"*Electrochimica Acta*, Vol. **51**, pp. 146-153, (2005).

[20] B. Chi, J. Li, X. Yang, Y. Gong and N. Wang, "Deposition of Ni–Co by Cyclic Voltammetry Method and Its Electrocatalytic Properties for Oxygen Evolution Reaction,"*International Journal of Hydrogen Energy*, Vol. **30**, (2009).

[21] B. Tury, M. Lakatos-Varsanyi and S. Roya, "Ni–Co Alloys Plated by Pulse Currents," *Surface &CoatingsTechnology*, Vol. **200**, pp. 6713-6717, (2006).

[22] W. E. G. Hansal, B. Tury, M. Halmdienst, M. L. Varsanyi and W. Kautek, "Pulse Reverse Plating of Ni–Co Alloys: Deposition Kinetics of Watts, Sulfamate and Chloride Electrolytes," *ElectrochimicaActa*, Vol. **52**, pp. 1145-1151, (2006).

[23] V. D. Jovic, B. M. Jovic and M. G. Pavlovic, "Electrodeposition of Ni, Co and Ni–Co Alloy Powders," *ElectrochimicaActa*, Vol. **51**, pp. 5468-5477, (2006).

[24] V. D. Jovic, B. M. Jovic, V. Maksimovic and M. G. Pavlovic, "Electrodeposition and Morphology of Ni, Co and Ni–Co Alloy Powders Part II. Ammonium Chloride Supporting Electrolyte," *ElectrochimicaActa*, Vol. **52**, pp. 4254-4263, (2007).

[25] A. Dolati, M. Sababi, E. Nouri and M. Ghorbani, "A Study on the Kinetic of the Electrodeposited Co–Ni Alloy Thin Films in Sulfate Solution," *Materials Chemistry andPhysics*, Vol. **102**, pp. 118-124, (2007).

[26] R. Orinakova, A. Oriňák, G. Vering, I. Talian, R. M. Smith and H. F. Arlinghaus, Influence of pH on the Electrolytic Deposition of Ni–Co Films,"*Thin SolidFilms"*, Vol. **516**, pp. 3045-3050, (2008).