

EFFECT OF TEMPERATURE AND CURRENT DENSITY IN ELECTRODEPOSITED Co-W MAGNETIC NANO THIN FILM

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Co-W thin films were prepared by electrodeposition with tri sodium citrate as organic additives. Normally Co-W alloys having high hardness. Its hardness value was studied in different current density and temperature. Co-W film having hard magnetic property at low temperature range and it is slightly modified to soft magnetic character when the temperature is increased to higher level. It was studied by using VSM. Surface morphology was studied by using XRD and SEM measurement.

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1. Introduction

The electrodeposition of alloys has been gaining in importance in recent years owing to its practical utility in modern technology for special items which cannot be produced in any other way. There are many different methods to deposit the film on the substrates but electrodeposition is the cheapest, low cost and easy method. Recent efforts have been directed to preparing magnetic alloys which are of great interest in the sophisticated electronics industry related to rocketry, computers, space technology etc. [1-4] In the fastest world thin and low cost systems are dominated, like MEMS and NEMS were developed with high efficient. The hard and soft magnetic materials are used in MEMS and NEMS including micro-actuators, sensors, micrometers and frictionless microgears [5-7]. The Soft magnetic thin films are used in recording head and hard magnetic films are used in storage devices [8-9].

The Ni, Co & Fe alloys are electroplated directly and uses for different magnetic application. Electrodeposition method is mainly used for the application of micro fabrication technologies. Tungsten has not deposited directly as a pure metal from the aqueous solution but co-deposit with other metal. Many of the researchers focus to the iron group alloys are electrodeposited with tungsten because of high crystalline temperature and corrosion resistance [10]. Cobalt layers can be obtained with different microstructures by deposition depending mainly electrolytes, solution pH, temperature and deposition current density (c.d.) [10-12]. Tungsten is co-deposited with cobalt to make hard alloy film with high tensile and corrosion resistance magnetic film.

The purpose of current study is the deposition of cobalt- tungsten alloy from tri sodium citrate of sodium electrolyte and to change the temperature and current density.

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2. Experimental procedure

A copper substrate of size 1.5X 5 c.m as cathode and platinum plate is used as anode for galvanostatic electrodeposition method. A d.c current for electrodeposition was passed from a regulated power supply. Analytical grade chemicals were used to prepare bath solution. An adhesive tape was used as mask for all the substrate expect the area on which depositon of film was desired. The copper electrode was buffed for removing scratches in a mechanical polishing wheel using a buffing cloth with aluminum oxide abrasive. After buffed the substrates were cleaned by con H₂SO₄ or acetone. Before electrodeposition, these substrates were electro cleaning in an alkaline electro cleaning bath after that the substrates were rinsed in distilled water. Electrodeposition was done by different current density and temperature.

Electrodeposition of cobalt-tungsten magnetic thin film were plated from a bath contained (CoSO₄.7H₂O) 0.1 M, sodium tungstate (Na₂WO₄.2H₂O) 0.05 M, sodium sulphate (Na₂SO₄) 0.3M, and tri sodium citrate (Na₃C₆H₅O₇.2H₂O) 0.3M. The pH of the path was maintained at 8.0 and the temperature ranging as 40 & 60°C. The thin film was coated for different time like 15 min, 30 min & 45 min. The properties of cobalt – tungsten were investigated.

The thickness of the deposits was tested using digital micrometer (Mitutoyo , Japan). Magnetic properties of deposited films were studied using vibrating sample magnetometry. In this technique the material under study was contained in a sample holder, which was centered in the region between the pole pieces of a laboratory magnet. A slender vertical sample rod connects the sample holder with a transducer assembly. The transducer converts a sinusoidal alternating current drive signal into a sinusoidal vertical vibration of the sample rod. Coils mounted on the pole pieces of the magnet pick up the signal resulting from the sample motion. X-ray diffractometry (XRD) Rich Seifert, Germany of model 3000 and scanning electron microscopy (SEM) Mosumy Electronics Japan make JEOL were used to study the structure and morphology of these magnetic films respectively. “From XRD data crystallite size of the deposited CoW and film stress was calculated. Percentage of elemental analysis of CoW film was used EDAX. Hardness of the deposit was obtained using vicker hardness tester using diamond intender method. Adhesion of the film was tested by bend and by scratch or chisel test. These tests are widely used in the field of electroplating.

3. Results and discussion

3.1 Thickness Study

Thickness of the film was increased with increase in current density as well as increase in duration of deposition for the temperature 40 and 60°C. It shows from the table.1.

Table.1. the thickness and magnetic properties of Co-W film from the different temperature and current density.

Temp (°C)	Current density (mA/cm ²)	Deposition time (min)	Thickness of the deposit (µm)	Magnetic saturation (emu)	Remanent (emu)	Coercivity (Oe)	Squareness
40	20	15	0.3	0.969	0.32	831	0.33
		30	0.6	0.954	0.31	792	0.33
		45	0.8	0.946	0.29	760	0.31
	25	15	0.5	0.932	0.26	722	0.28
		30	0.9	0.920	0.24	690	0.26

Temp (°C)	Current density (mA/cm ²)	Deposition time (min)	Thickness of the deposit (μm)	Magnetic saturation (emu)	Remanent (emu)	Coercivity (Oe)	Squareness	
		45	1.3	0.910	0.21	650	0.23	
		30	15	0.8	0.901	0.29	629	0.32
			30	1.2	0.887	0.25	605	0.28
			45	1.7	0.873	0.22	580	0.25
60	20	15	0.5	0.864	0.28	565	0.32	
		30	1.0	0.850	0.26	524	0.31	
		45	1.6	0.839	0.21	500	0.25	
	25	15	0.9	0.823	0.25	472	0.30	
		30	1.4	0.810	0.22	450	0.27	
		45	1.9	0.800	0.19	419	0.24	
	30	15	1.4	0.787	0.24	396	0.30	
		30	1.8	0.771	0.20	367	0.26	
		45	2.5	0.762	0.18	340	0.23	

Table.2. Crystalline size, hardness and composition of Co-W films for different temperature and current density.

Temp (°C)	Current density (mA/cm ²)	Crystalline size (nm)	Vicker Hardness (VHN)	Film Composition (at%)	
				Co	W
40	20	80	580	75.74	24.26
	30	56	628	72.5	27.5
60	20	64	595	73.53	26.47
	30	45	646	70.7	29.3

3.2 Surface characterization

X-ray diffraction patterns of Cobalt –tungsten electrodeposits were produced from different temperature bath like 40 and 60°C for 20 and 30 mA /cm² and 30 min were fixed the current density and time of deposition respectively for electrodeposition . The data obtained from the XRD pattern compared with standard data and were found to have hexagonal close packed structure and exhibited (200) plane predominantly. From the XRD pattern peak, stressed in the film was calculated using the formula:

$$\text{Youngs modulus} = \text{stress/strain.}$$

Crystalline sizes of the deposits were calculated from the XRD pattern using the formula: Crystalline size = $0.9\lambda/B\cos\theta$.

These values clearly show that the crystallite sizes of the Co-W deposit obtained by electrodeposition process are in the nano scale. The crystallite sizes of deposits are shown in Table 2.

From XRD data the crystalline structure found in Co₃W film is produced hcp from a bath at low temperature and the crystalline size is decreased by increasing the temperature during electrodeposition. The films are high hardness and good adhesion.

Electrodeposited cobalt -tungsten films from different temperature bath as mentioned in XRD studies were subjected to SEM studies. The micrographs are shown in Fig. 1. In general, microstructure of the Co-W film is greatly influenced by the temperature of bath. Elements present in the film were analyzed by Energy dispersive X-ray spectroscopy and the results are shown in Table 2

3.3 Mechanical properties

Adhesion of the film with the substrate is tested by bend test and scratch test. It showed that the film is having good adhesion with the substrate. Hardness of the film increases when the film deposited from the temperature and current density increases. The results are reported in Table 2.

3.4 Magnetic properties

Electrodeposition studies were carried out using different bath temperature. Table 2 shows the results of electrodeposition of Co-W and their magnetic properties for different bath temperatures like 40 and 60 °C. When the low temperature bath was used for deposition, the thickness of the deposit increases with increase in current density and time of deposition. Films are having high coercive and remanent values. At low temperature the film acts as hard magnetic property.

In the electrodeposition studies films produced (60°C) were uniform and bright. This because the orientation of crystallization uniformly during electrodeposition by adsorbing itself on the initially deposited crystals. The magnetic properties of the high temperature bath films revealed that these films are having a low coercive and remanent value when compared to the deposit obtained from films deposited from low temperature bath.

As the average crystallite sizes of the films are in nano scale, considerable change in magnetic behavior can occur. When the crystalline size approaches low nano level, the domain wall thickness is comparable to the crystalline size the coercivity found to low. Analysis of crystallite size, microstructure and magnetic properties confirm that the origin of magnetic properties is because of the strongly interacting array of single domain crystals. This is mainly due to the films from high temperature bath

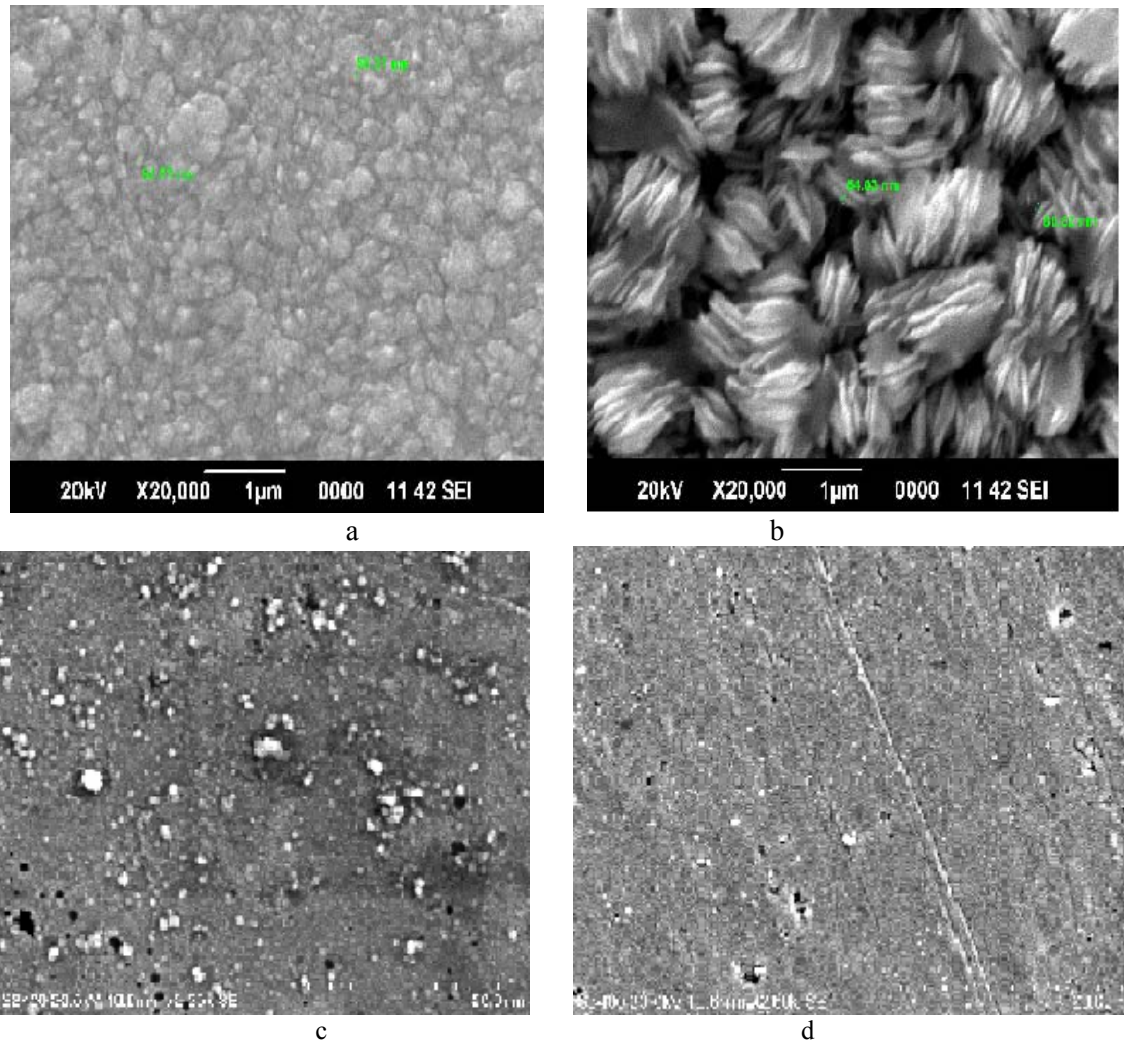


Fig. 2. SEM images (a)20mA,(b)30mA at 40°C &(c)20mA(d)30mA at 60°C.

4. Conclusion

A cobalt-tungsten film having good hard magnetic properties can be electrodeposited from the low temperature bath containing percentage of tungsten is slightly increased. In the high temperature bath the film character is change it showed soft magnetic character. It also increases the film stress, which is a cause for cracked film. The high temperature bath films are lower stress which are used in MEMS devices. Hardness of the film also increases in higher temperature bath films which are having lower coercivity value and lower remanent values. Also these films have good adhesion with the substrate and their crystalline sizes are nano scale.

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