Effect of bath temperature on structural and magnetic properties of electrodeposited NiFeCr thin films

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The nano crystalline NiFeCr thin films were synthesised on the surface of aluminium substrate at different bath temperature like 30, 40, 50 and 60˚C by applying a constant current density of 0.5 A/dm^2. All the coated NiFeCr thin films were subjected to the structural, mechanical and magnetic characterization analysis (EDAX, SEM, XRD, VHT and VSM). The SEM pictures of NiFeCr thin films shows that, the film have crack free, uniform and bright surface morphology with fine grain size. The XRD pattern reveals the BCC structure of NiFeCr thin films with three predominant diffraction peaks. The VSM data of NiFeCr clearly shows their magnetic nature. The NiFeCr thin films coated at high bath temperature have highest saturation magnetisation value with lower coercivity. Because of its low coercivity with highest magnetisation value, the NiFeCr thin films may used for the manufacturing of MEMS devices.

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1. Introduction

Magnetic thin films are the most commonly required materials for various applications such as magnetic writing heads, MEMS devices, Corrosion resistance and sensors. Normally NiFe thin films have best magnetic properties like low coercivity, high saturation magnetisation, high permeability and nearly zero magnetostriction. Mostly NiFe thin films are the best known soft magnetic thin films and addition of Mn, Co, Mo, Al and Cr to NiFe films may alter the structural and magnetic properties. So in this present work, we planned to examine the magnetic properties of NiFeCr thin films. There are number of physical and chemical methods available for the synthesis of magnetic thin films. Among the various methods, electrodeposition is the suitable method for the synthesis of magnetic thin films. Because electrodeposition method variety of advantages compared with other physical and chemical methods such as low cost, large scale production and easy to monitoring the film deposition rate etc.,

This investigation summarises the synthesis and magnetic properties of electrodeposited NiFeCr thin films coated at different bath temperature. Reason for the changes in physical and magnetic properties was also discussed.

2. Experimental part

The bath composition and working conditions of electrodeposited NiFeCr thin films are shown in the table.1. The NiFeCr thin films are electrodeposited at various bath temperatures of 30,40,50 and 60^{0}C. The aluminium substrate is used for both anode and cathode (7.5 x 1.5 cm). The aluminium substrate (cathode) is covered with adhesive tape except the area of deposition is required. Before the electrodeposition process, the substrates were polished with silicon carbide emery paper and degreased with 1M of NaOH for 5 minutes then rinsed with double distilled water and dried in air. The pH of the bath is maintained as 3. The films were coated on the cathode surface at various temperatures from 30^{0}C to 60^{0}C. All the NiFeCr thin films were coated at constant current density of 0.5 A/dm^2 and constant deposition time of 30 minutes. The surface morphology and structure of the deposited NiFeCr thin films were analysed with The Scanning Electron Microscopy (SEM) and X-ray diffraction (XRD). Vibrating Sample Magnetometer (VSM) is used to determine the magnetic properties. The compositions of the films were studied by using Energy-dispersive X-ray Spectroscopy (EDAX) analysis. Vickers hardness tester (VHN) is used to determine the hardness of the deposited film.

Table 1. Chemical composition and operating conditions of the electroplating bath.

Bath chemicals (g/L)		Temperature (^{0} C)	pH	Current density (A/dm^2)
Nickel chloride	80	30,40,50,60	3	0.5
Ferric chloride	65			
Chromium chloride	50			
Boric acid	30			
Glycine	50			
Ammonium formate	30			

3. Results and Discussion

3.1 Composition of deposits

The chemical composition of NiFeCr thin films are obtained by EDAX analysis and it is shown in table 2. The films coated at bath temperature of 30˚ C have Ni content of 57.78 wt% ,Fe content of 41.21 wt% and Cr content of 1.01 wt%. The electrodeposited bath temperature was increased from 30˚C to 60˚C. The Ni content of the films is gradually increased and NiFeCr films coated at 60˚ C have highest Ni content of 74.08 wt%. The corresponding Fe content was decreased while increasing the bath temperature. From EDAX analysis we concluded that the NiFeCr thin films coated at 60˚ C have highest Ni content with Cr content of 2.60 wt%.

Table.2 Results of EDAX analysis

S. No	Bath Temperature (^{0}C)	Ni wt %	Fe wt %	Cr wt %
1	30	57.78	41.21	1.01
2	40	62.93	35.81	1.26
3	50	69.90	28.97	1.13
4	60	74.08	23.33	2.60

3.2 Morphological observation

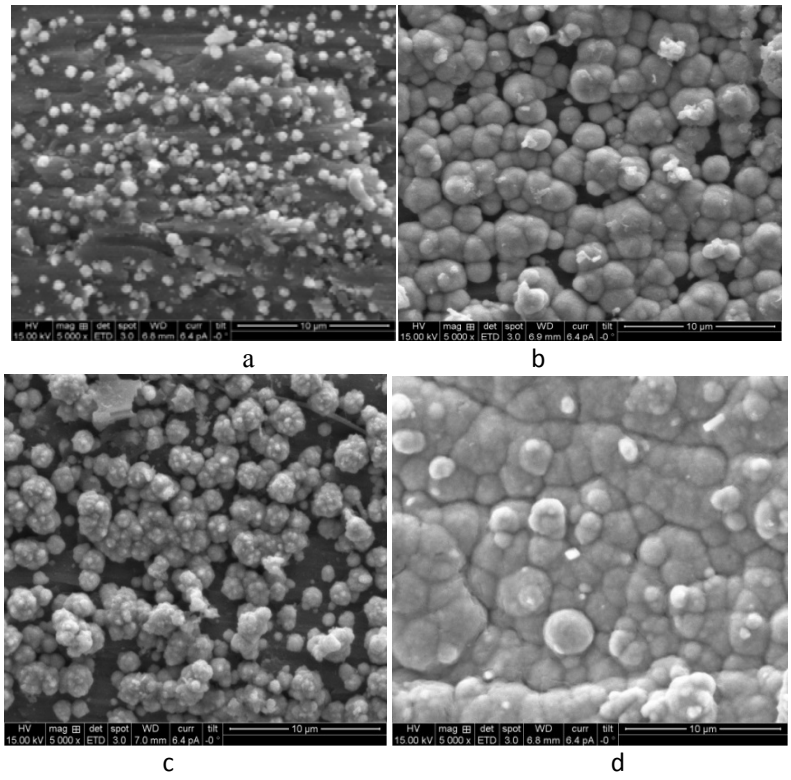

Fig.1. SEM images of NiFeCr films electrodeposited at (a) 30^{0}C (b) 40^{0}C (c) 50^{0}C (d) 60^{0}C

The surface morphologies of the coated NiFeCr films were studied with SEM pictures as shown in fig.1. The deposited films have uniform surface morphology with fine grain structures. The SEM pictures of NiFeCr thin films are clearly shows that the NiFeCr films are crack free. In the NiFeCr film coated at 60˚ C, the grains are clearly visible and have uniform surface.

3.3 Structural analysis

Fig. 2 shows the XRD patterns of NiFeCr thin films coated at different bath temperatures from 30^0C to 60^0C. The obtained XRD data of NiFeCr thin films are compared with standard JCPDS data and found to have BCC structure. The peaks are found at (110), (200) and (211) for all the coated NiFeCr thin films.

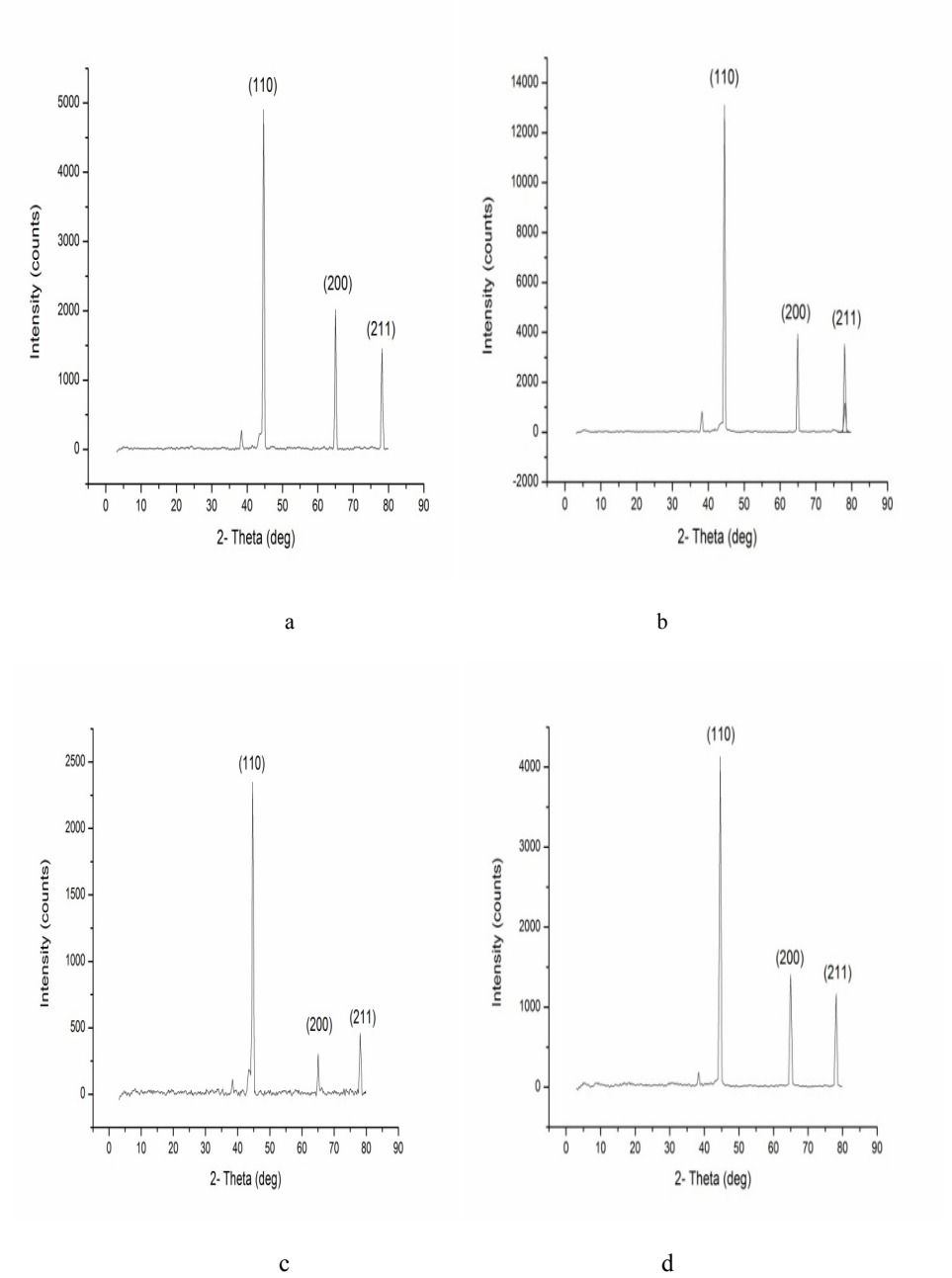

Fig.2 XRD patterns of NiFeCr thin films (a) 30^0C (b) 40^0C (c) 50^0C (d) 60^0C

The crystalline size of the coated NiFeCr thinfilms was tabulated as shown in table.3. The electrodeposited NiFeCr thin films have crystalline nature. The crystalline sizes of the coated thin films are calculated by using Scherrer's formula

$$D = \frac{0.9\lambda}{\beta cos\theta} \quad (1)$$

Where, λ is the X-ray wavelength, β is the full width at half maximum intensity of the diffraction peak located at 2θ and θ is the Bragg's angle. These values clearly shows that the crystalline size of NiFeCr thin films are in the nano scale. The average crystalline size of NiFeCr thin films is found to have 28.5 nm. The crystalline sizes are gradually decreased while increasing the bath temperature. The film coated at 60˚ C have lowest crystalline size of 24.6 nm as compared to the film coated at 30˚ C. From XRD, it is concluded that the crystalline size decreases while increasing the bath temperature. This is due to onset of crystals during electrodeposition.

Table.3 Structural characteristics of NiFeCr thinfilms

S. No	Temperature (^{0}C)	2□ (deg)	□ (deg)	Particle size, D (nm)	Strain (10^{-3})	Dislocation density (10^{14} / m^2)	d (A^0)	B (10^{-3})
1	30	64.900	32.450	31.906	1.1405	9.823	2.03	5.40
2	40	44.642	22.321	30.381	1.1979	10.834	2.02	5.18
3	50	44.668	22.334	27.592	1.1319	13.135	2.02	5.70
4	60	44.582	22.291	24.645	1.4765	16.464	2.03	6.38

3.4 Mechanical properties

The hardness of the coated films was tested by using diamond intended method by using Vickers hardness tester as shown in table.4. The hardness of the film slightly increases as the temperature increases.

Table.4 Mechanical properties of electrodeposited NiFeCr thinfilms

Temperature (^{0}C)	Vickers hardness(VH)	Thickness(μm)
30	62.7	3.048
40	65.8	7.226
50	66.2	7.292
60	69.5	4.178

3.5 Magnetic properties

The magnetic properties of the films are strongly depends on the nano crystalline nature of the film. The magnetic properties of NiFeCr thin films were analysed by using VSM as shown in table.5. The VSM patterns of electrodeposited NiFeCr thin films are shown in Fig.3. The NiFeCr thin films coated at room temperature have coercivity of 88.7 Gauss with saturation magnetisation of 68.395 x 10^{-3}emu/cm^2. The films coated at 60˚ C have highest magnetisation value of 1.605 emu/cm^2 with corresponding coercivity value of 45.041 Gauss.

Table.5 magnetic properties of the NiFeCr films

S.No	Temperature (^{0}C)	Coercivity G	Magnetisation M_s (emu/cm^2)	Retentivity M_r x 10^{-3} (emu/cm^2)	Squareness S (M_r/ M_s)
1	30	88.702	68.395 x 10^{-3}	6.9622	0.10179
2	40	86.905	0.2013	21.282	0.10572
3	50	61.750	0.474	27.505	0.05802
4	60	45.041	1.605	86.974	0.05417

From table 5 it is concluded that the coercivity of the NiFeCr films decreases while increasing the bath temperature to 60˚ C and their magnetisation values are increased to high value. The NiFeCr films coated at high temperature exhibit the best soft magnetic nature (low coercivity with high magnetisation). This is due to the occurrence of nano crystalline phase at high bath temperature as compared to low bath temperature. Because magnetic properties are strongly affected by crystalline size of the film. The temperature also affects the magnetic nature of the coated film. The highest saturation magnetisation is 1.605 emu/cm^2 with 45.041 Gauss of coercivity is due to the highest Ni content and the presence of Cr content in electrodeposited NiFeCr thin films.

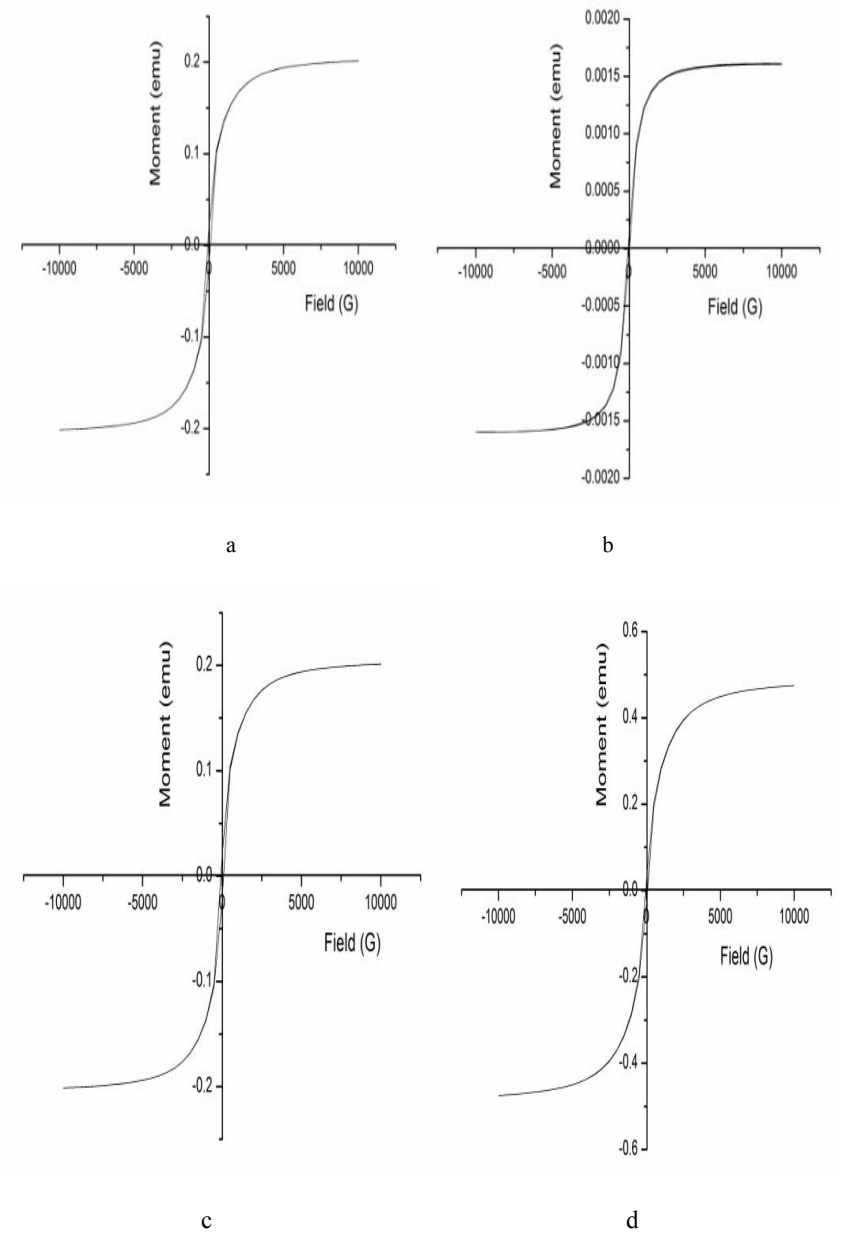

Fig.3 Hysteresis loops of NiFeCr films electrodeposited at (a) 30^{0}C (b) 40^{0}C (c) 50^{0}C (d) 60^{0}C

4. Conclusion

Nano crystalline NiFeCr electroplated thin films have been successfully coated on Al substrate. The structural and magnetic behaviour of NiFeCr thin films were properly analysed. The result shows that,

1. The electroplated NiFeCr thin films have average crystalline size of 28.5 nm and the films have BCC structure with three diffraction peaks.

2. NiFeCr coatings have uniform and bright surface morphology. All the coated films are free from micro cracks and voids.

3. While increasing the bath temperature from 30 ˚ C to 60 ˚ C, the Ni content of NiFeCr thin films increased from 57.78 wt% to 74.08 wt% and the Fe content was decreases while increasing the bath temperature.

4. The NiFeCr thin films have good adherent with substrate and Vickers hardness of the film is low as 66 VHN.

5. The NiFeCr thin films coated at room temperature (30 ˚ C) have coercivity of 88.7 Gauss with saturation magnetisation value of 68.395×10^{-3} emu/cm^2.

6. The NiFeCr thin films coated at

60˚C have coercivity of

45.04 Gauss with saturation magnetisation value of 1.605 emu/cm^2.

7. The VSM analysis clearly shows that the coercivity of NiFeCr thin films decreases while increasing the bath temperature and the corresponding magnetisation values were increased to high value.

8. The electroplated NiFeCr thin films coated at high temperature exhibits the best magnetic behaviour and these films may used in MEMS area.

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