Impact of Additive on Magnetic Properties of Nano Crystalline NiFeCr Thin Films

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***Abstract* :- In this investigation, the nano crystalline NiFeCr thin films were successfully coated by electrodeposition method by varying the bath temperature from 30 to 50º C which contains the additive like sodium hypo phosphite. The current density and bath pH values have been kept constant as 0.5 A/dm^2 and 3. Electroplated NiFeCr thin films were subjected to morphology, structural and magnetic studies. The chemical composition of the films was determined from EDAX results. The SEM picture reveals that, the film have bright ,uniform and crack free surface morphology. The X-ray diffraction studies of NiFeCr thin films shows the crystalline nature with BCC sturcture and the film have average particle size of 37 nm. Sodium hypo phosphite influences the magnetic properties of NiFeCr thin films. At 50 ° C, the film have highest saturation magnetisation with lower coercivity . This is due to the presence of additive in the electroplating bath. While increasing the bath temperature from 30 to 50º C , the magnetisation values were increased. The reasons for the variation of magnetic properties were also discussed**

Key words :- Additive, sodium hypophosphite, magnetic properties , bath temperature and coercivity.

1. INRODUCTION

NiFe based thinfilms have wide applications in magnetic recording media, memory chips, sensors etc., [1-6]. The high magnetic saturation with low coercivity, high permeability and high electrical resistivity [7-10] are the essential requirements for the NiFe based thinfilms in the field of MEMS and magnetic recording heads[11-15]. Among the various physical and chemical deposition methods electrodeposition method is suitable for NiFe based thinfilms. The grain size and growth orientation are the dominant factors to decide the magnetic and mechanical properties of NiFe based thinfilms. These factors may be affected by current density, bath temperature and pH value etc,. In this research work optimised current density for NiFeCr thinfilms were already found. Because of this suitable current density and bath temperature the coated films have grain size is in the range of nano scale. The roles of various salts and additives on NiFe based thinfilms have been investigated over the century. The main focus of the present work is the enhancement of magnetic properties of NiFeCr thinfilms by adding the sodium hypophosphite. The NiFeCr thin films coated with sodium hypophosphite bath have been compared with the NiFeCr thinfilms coated without additive.

2. EXPERIMENTAL PART

The bath composition and working parameters of electrodeposited NiFeCr thin films are tabulated in the table 1. The NiFeCr thin films are electrodeposited from the bath which contains the additive (sodium hypophosphite) at temperatures 30, 40 and 50 ^{0}C with time period of 30 minutes. The pH value of the solution is maintained as 3. Both the anode and cathode (7.5 x 1.5 cm) are the aluminium substrates. The cathode substrate is covered with adhesive tape except the desired area of deposition. Before the electrodeposition, 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 current density is 0.5 A/dm^2 for all the coated NifeCr thin films. The surface morphology and micro structure of the electrodeposited NiFeCr thin films were analysed with the Scanning Electron Microscopy (SEM) and X-ray diffraction (XRD). Vibrating Sample Magnetometer (VSM) is used to analyse the magnetic properties.

The compositions of the thinfilms 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. The crystalline size of the coated films were calculated by using the Scherrer's formula

$$D = \frac{0.94\,5\lambda}{\beta cos\theta} \qquad (1)$$

The strain (ε) was calculated using the relation

$$\varepsilon = \frac{\beta\, cos\theta}{4} \qquad (2)$$

Table 1. Bath composition and operating conditions of the electrodeposition bath with additive

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

3. RESULTS AND DISCUSSION

3.1 Composition of deposits

The chemical composition of NiFeCr thinfilms were analysed by using EDAX analysis are shown in table 2.

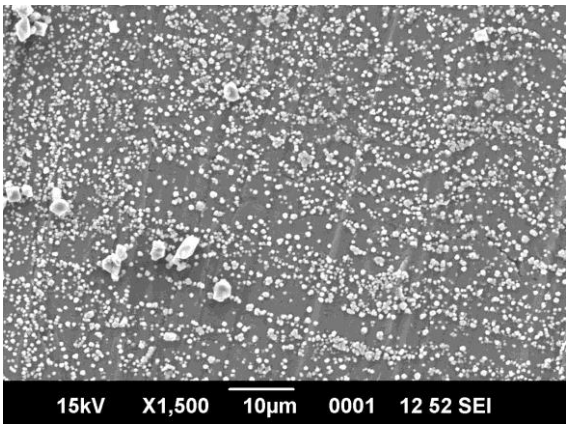
Table.2 EDAX analysis of NiFeCr thin films coated from sodium hypophosphite.

S.No	Temperature (^{0}C)	Ni wt %	Fe wt %	Cr wt%
1	30	75.97	21.65	2.38
2	40	80.81	15.26	0.94
3	50	81.24	18.53	0.22

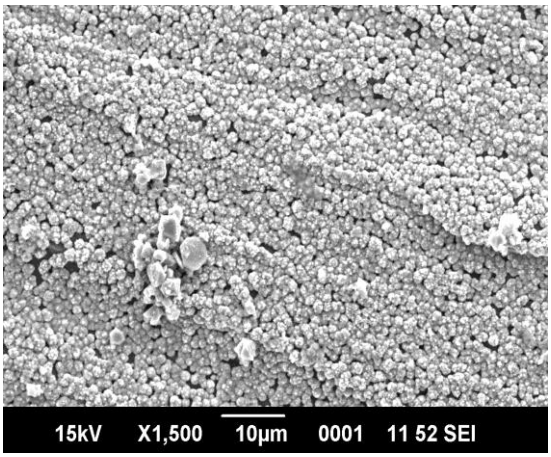
The EDAX results showed that, the wt % of Ni increasing with increasing the bath temperature. The corresponding wt % of Fe and Cr has been decreased with increasing the bath temperature. The sodium hypophosphite influences the chemical composition of NiFeCr thin films. The film coated at 50 ^{0}C have highest Ni content of 81.24% . This result was compared to NiFeCr thinfilms coated from without additive. The addition of sodium hypophosphite to electrodeposited bath increases the Ni content and decreases the Cr content. Because of the high Ni content with lower Cr content, NiFeCr films may have enhanced saturation magnetization.

3.2 Surface Morphology of the NiFeCr thin films

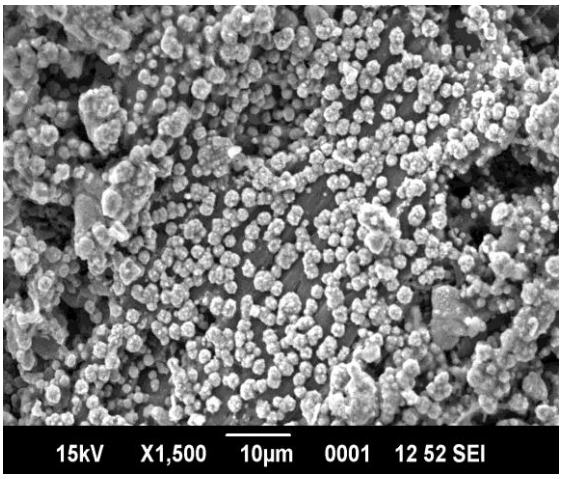
The surface morphology of fabricated NiFeCr thinfilms were analyzed with the help of Scanning Electron Microscope(SEM). The SEM images of the electrodeposited NiFeCr thinfilms are shown in fig.1. The deposited films are bright, uniform and crack free surfaces. The film coated at 50^{0}C have bright and smaller crystallites. This is may be due to the addition of sodium hypophosphite.



(a)



(b)

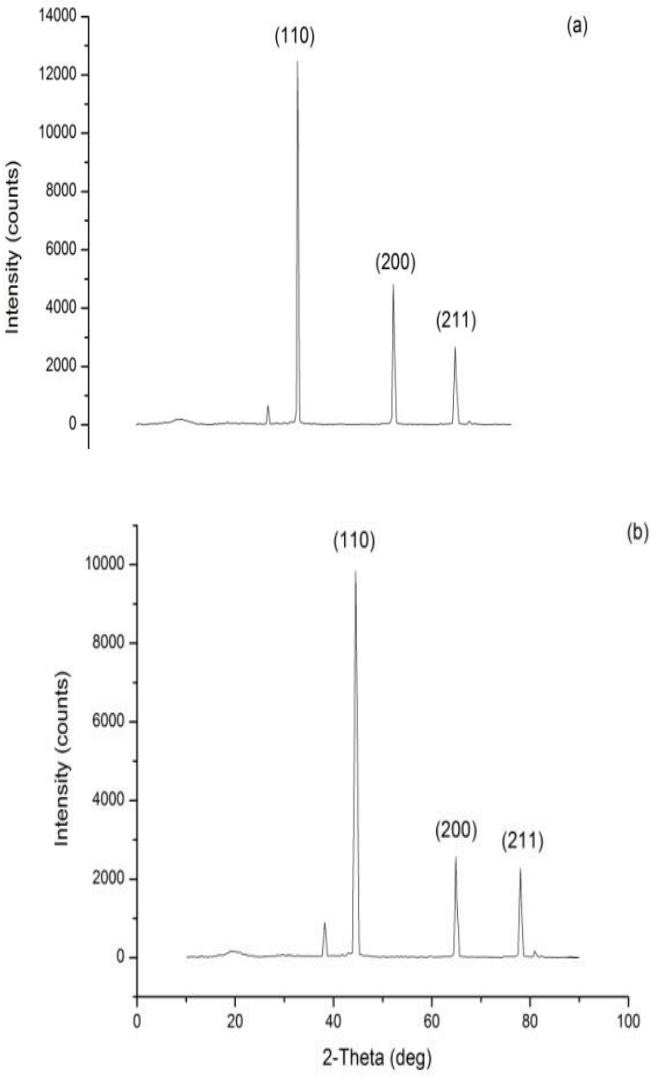


(c)

Fig.1 SEM images of NiFeCr films coated from sodium hypophosphite bath at (a)30 ^{0}C (b) 40 ^{0}C (c)50 ^{0}C

3.3 Structural Analysis of electroplated NiFeCr thin films.

The NiFeCr thin films coated at various temperatures are subjected to XRD analysis are shown in fig 2.



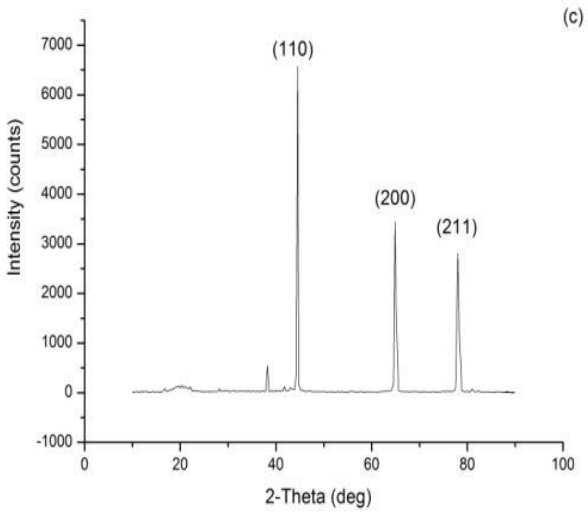


Fig.2 XRD patterns of NiFeCr thin films

The presence of sharp peaks shows the crystalline nature of NiFeCr thinfilms. The XRD patterns of NiFeCr reveals the existence of BCC structure with diffraction peaks at (110), (200) and (211) . and found to have BCC structure. The average particle size of NiFeCr thin films were found to have 37 nm. The films coated at high temperature have the smallest grain size. The sodium hypophosphite and bath temperature may reorient the crystallites during the electrodeposition process.

Table.3 Structural characteristics of NiFeCr thinfilms

S.No	Temperature (^{0}C)	2θ (deg)	θ (deg)	Particle size, D (nm)	Strain (10^{-4})	Dislocation density (10^{14}/m^2)	d (A^0)	β (10^{-3}) (radian)
1	30	44.490	22.245	42.533	8.557	5.528	2.0357	3.698
2	40	44.463	22.231	37.259	9.768	7.203	2.0347	4.221
3	50	44.488	22.244	29.959	12.15	11.14	2.0347	5.250

3.4 Mechanical property

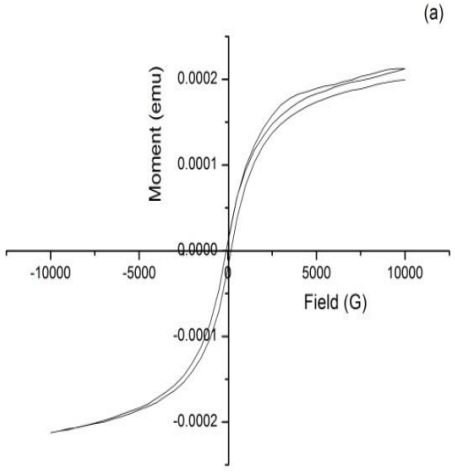
Micro hardness of electroplated NiFeCr thin films were analysed by using Vickers hardness method. All the coated films have good adherent with substrate. This was observed by bend and scratch test. The hardness of the NiFeCr thin films are in the low range which is shown in the table 4.

Table.4 Mechanical properties of electrodeposited NiFeCr thinfilms

Temperature (^{0} C)	Vickers hardness(VHN)	Thickness (μm)
30	265	1.3055
40	190	2.4333
50	184	2.4638

3.5 Magnetic Properties

The VSM graphs of NiFeCr thinfilms are shown in figure 3. The magnetic properties are mainly decided by parameters like saturation magnetisation, coercivity, retentivity and crystalline size.



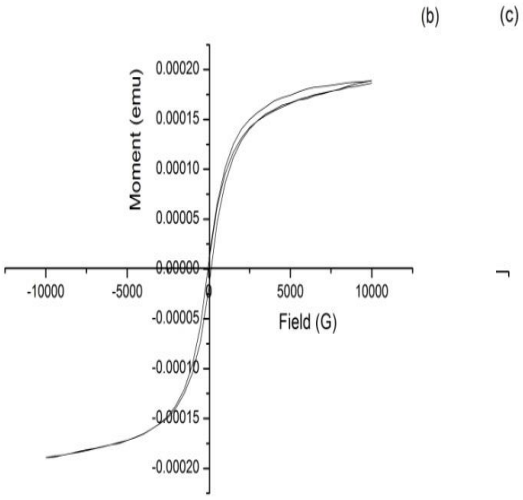


Fig.3 Hysteresis loops of NiFeCr films from bath containing sodium hypophosphite coated at (a)30 ^{0}C (b) 40 ^{0}C (c)50 ^{0}C

Table.5 magnetic properties of the films with varying temperature

Temperature (^{0}C)	Coercivity (G)	Magnetisation M_s 10^{-3} emu	Retentivity M_r 10^{-6} emu	Squareness S 10^{-3} (M_r/ M_s)
30	100.65	189.05	13.045	0.0690
40	137.09	212.58	15.641	0.0735
50	82.492	485.76	33.708	0.0693

The NiFeCr thin films coated from electrodeplated bath with additive at 50 ^{0}C have the saturation magnetization of 485.76 × 10^{-3} emu with coercivity of 82.492 Gauss. From table 5 it was observed that, The magnetization values were increased with increasing the sodium hypophosphite bath temperature. The results obtained for NiFeCr thinfilms were compared with NiFeCr thin films coated from the bath without additive. The addition of sodium hypophosphite greatly increases the saturation magnetization with lower coercivity . The films coated at lower temperature also have enhanced saturation magnetization values. The sodium hypophosphite greatly enhances the soft magnetic nature of NiFeCr thinfilms. Because of the enhanced magnetic properties, the NiFeCr thin films may be used in MEMS.

4. CONCLUSION

The impact of sodium hypophosphite on the structural, morphology and magnetic properties of electrodeposited NiFeCr thinfilms have been studied for varying temperatures from 30^{0}C to 50 ^{0}C. As the additive bath temperature increases the Ni content also increases. The highest Ni content of 81.24 with lower Cr content of 0.22 have been observed at 50 ^{0}C. From the SEM pictures we have observed that the films are crack free, bright and uniform. It is observed that the NiFeCr electrodeposits have high saturation magnetisation value with lower coercivity at 50 ^{0}C. Because of the best soft magnetic properties, the NiFeCr thin films may be used in MEMS and magnetic recording heads.

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