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Investigation of Structural and Magnetic Properties of Nano Crystalline NiFeCr Thin Films Electrodeposited from Sulphate Bath

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Abstract: In this study, the nano crystalline electrodeposited NiFeCr thin films were synthesised from sulphate bath on the surface of Al substrate by varying the current density from 0.5 A/dm^2 to 2 A/dm^2 at bath temperature of 30^0C. The effects of current density on structural, mechanical and magnetic properties of NiFeCr thin films have been investigated. The surface morphology of the deposits was studied by scanning electron microscopy (SEM) and chemical combination by energy dispersive X-ray spectroscopy (EDS). The crystalline nature of the film was analysed by using X-ray diffractometry (XRD). The magnetic properties such as coercivity, saturation magnetisation, retentivity, squareness of the NiFeCr deposits were studied by vibrating sample magnetometer (VSM) and the microhardness, thickness were also measured. From the XRD, the crystalline structure was found to be BCC and the average particle size is in the range of few tens of nanometers. The VSM result shows that the NiFeCr thin film coated current density of 2 A/dm^2 exhibits higher magnetisation of 91.370×10^{-3} emu with lower coercivity of 42.837 G. Because of the soft magnetic nature, NiFeCr thin films may be used in MEMS.
Keywords: Investigation, nano crystalline, micro hardness, current density, sulphate bath and coercivity.

Introduction

Electrodeposited soft magnetic thin films have potential applications in the field of data storage systems. In MEMS nano crystalline soft magnetic materials are widely used for the fabrication of micro switches, micro motors etc,. Among the various soft magnetic materials the NiFe based thin films are the best known materials for MEMS [1-3]. The magnetic properties like lower coercivity, higher saturation magnetisation and higher magnetic moment are the essential needed characters for MEMS devices [4-11]. Normally NiFe based alloys exhibit higher magnetisation with lower coercivity. In order to improve the reliability of magnetic storage devices Cr is decided to add with NiFe which have high corrosion resistance. This paper summarises the optimisation and effect of current density on structural and magnetic properties of NiFeCr alloys.

2. Experimental Part

The chemical composition and working conditions of the electroplated NiFeCr thin films are tabulated as shown in the table 1. The NiFeCr thin films are electrodeposited byvarying current densities from 0.5 A/dm^2 to 2 A/dm^{2}with deposition time of 30 minutes and controlled pH of 3. The temperature is maintained as 30 ^{0}C.

The aluminium plates act as both anode and cathode substrates which have size of 7.5cm x 1.5 cm. The cathode substrate is covered with adhesive tape except the area of deposition is required. 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 airjust before the electrodeposition. The surface morphology and micro structure of the electrodeposited NiFeCr thin films were studied with the Scanning Electron Microscopy (SEM) and X-ray diffraction (XRD). With the help of Vibrating Sample Magnetometer (VSM) the magnetic properties are analysed. The compositions of the thinfilms were determined by using Energy-dispersive X-ray Spectroscopy (EDS) analysis. Vickers hardness tester (VHN) is used to analyse the hardness of the coated film.

Byusing Scherrer's formula crystalline size of the coated films were calculated as

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

The strain (ε) was calculated from the following relation

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

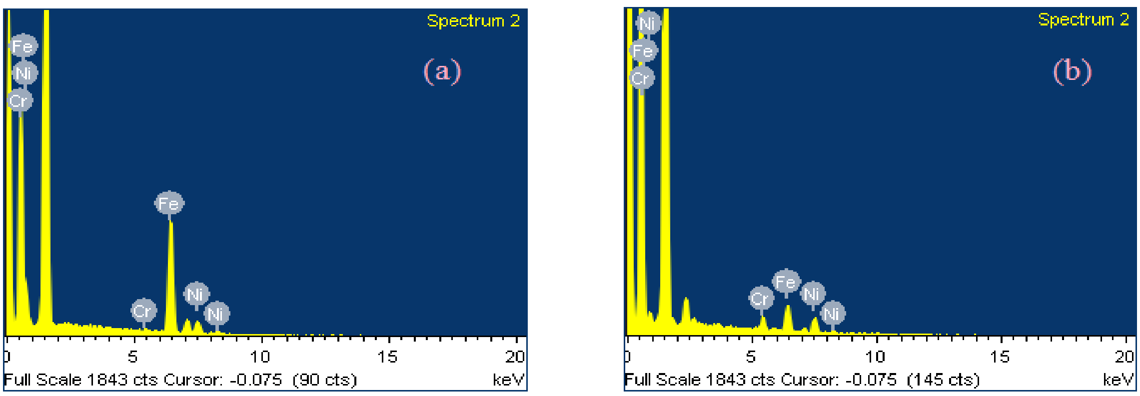
Table 1. Details of the electrodeposition bath.

Bath chemicals (g/L)		**Temperature (^{0}C)**	**pH**	**Current density (A/dm^2)**
Nickel sulphate	80	30	3	0.5, 1, 1.5, 2
Ferric sulphate	65			
Chromic sulphate	50			
Boric acid	30			
Glycine	50			
Ammonium formate	30			

3. Results and Discussion

3.1 Composition of deposits

All the synthesised NiFeCr thinfilms were subjected to EDAX analysis and the results are tabulated in Table 2.



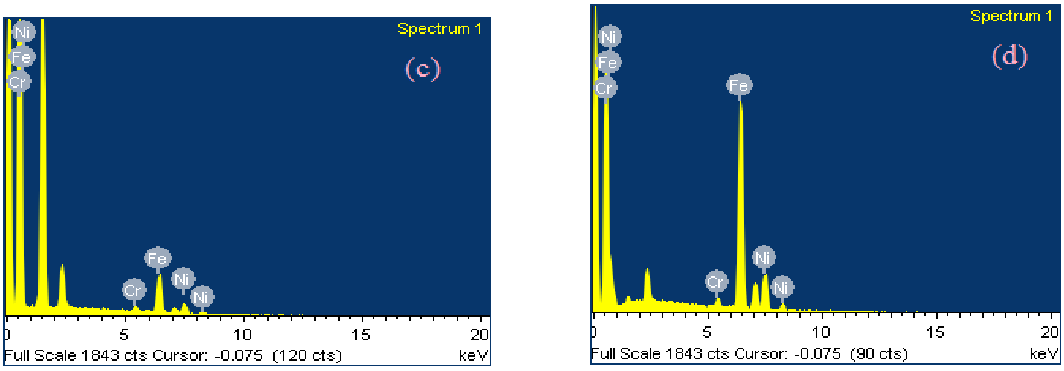


Fig.1 EDAX analysis of NiFeCr films electrodeposited at (a)0.5 A/dm^2 (b) 1 A/dm^2(c) 1.5 A/dm^2(d)2A/dm^2

The NiFeCr thin films coated from sulphate bath at current density of 2 A/dm^2 have lower Cr content of 0.96 wt % with nickel content of 14.28wt %. The Cr content was decreased while increasing the current density from 0.5 A/dm^2 to 2 A/dm^2. The corresponding Ni content was also decreased. Fe content has been increased from 46.43 wt % to 84.76 wt %.

Table 2. EDAX results of Electrodeposited NiFeCr films coated from sulphate bath

S.No	Current density (A/dm^2)	Ni Wt%	Fe Wt%	Cr Wt%
1	0.5	39.31	46.43	14.26
2	1	22.44	75.60	1.96
3	1.5	26.83	66.77	6.40
4	2	14.28	84.76	0.96

3.2 Surface Morphology of the deposits

The surface morphology of synthesised NiFeCr thinfilms for various current densities were studied with the help of Scanning Electron Microscope(SEM). Fig. 2 shows the SEM images of the electrodeposited NiFeCr thinfilms. The films deposited at lower current density are bright, uniform and crack free surfaces. The NiFeCr thin films coated at higher current density have fine ganular. This may be due to the reduction of internal stress during electrodeposition.

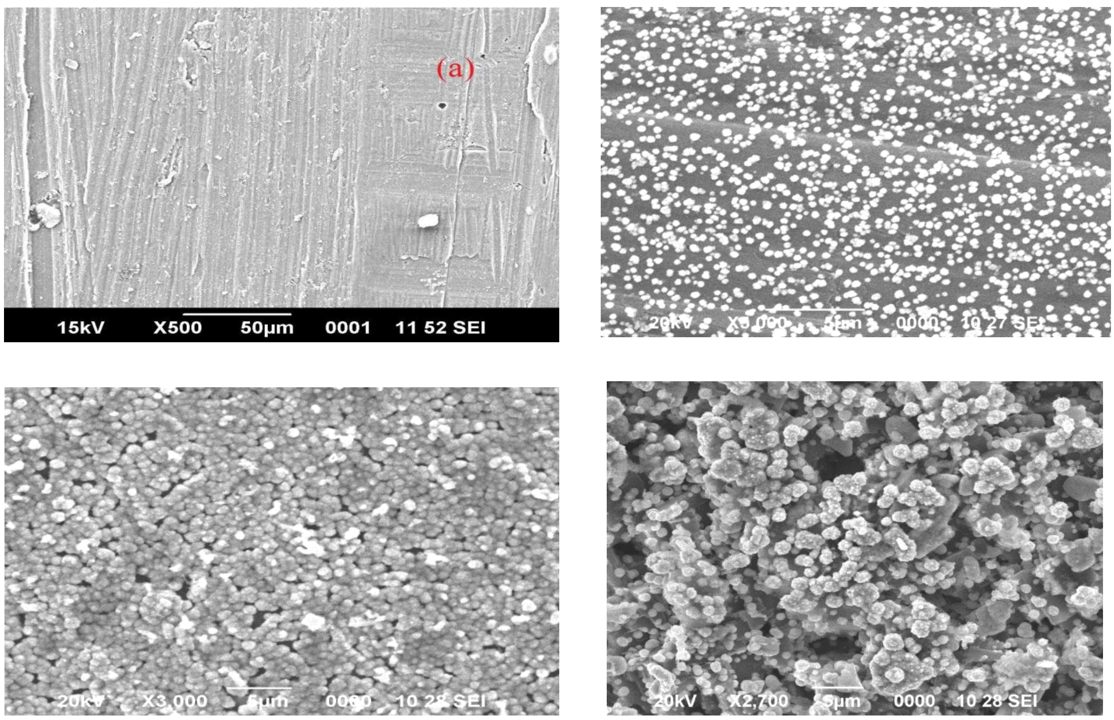


Fig.2. SEM pictures of NiFeCr films electrodeposited from sulphate bath at (a)0.5A/dm^2 (b)1A/ dm^2 (c) 1.5 A/ dm^2(d) 2A/ dm^2

3.3 Structural Analysis

The electrodeposited NiFeCr thin films from sulphate bath at different current densities are subjected to XRD analysis and are shown in fig. 3. All the coated films have sharp diffraction peaks at (110), (200) and (211). This is due to the crystalline nature of NiFeCr thin films. The crystallite size was calculated by using Scherrer's formula and it was found to be in the range of nano scale. The XRD result reveals that the NiFeCr thin films have BCC structure with average crystallite size of 39 nm.

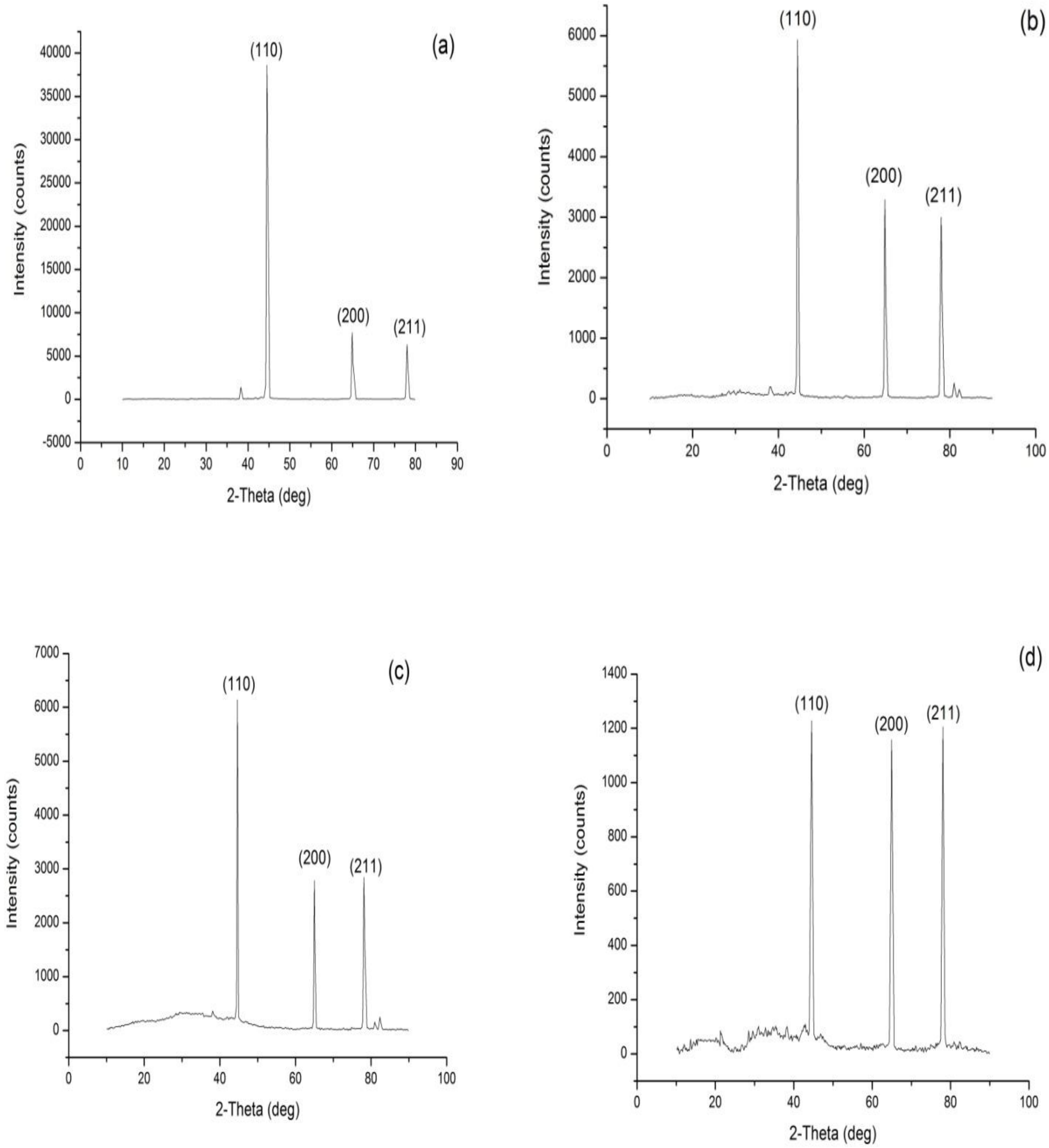


Fig.3. XRD patterns of NiFeCr films electrodeposited from sulphate bath at (a)0.5A/dm^2 (b)1A/ dm^2 (c) 1.5 A/ dm^2(d) 2A/ dm^2

Table.3 Structural characteristics of NiFeCr thinfilms

S.No	Current density (A/dm^2)	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	0.5	44.505	22.252	40.623	8.9625	6.0597	2.0342	3.873
2	1	44.470	22.235	41.739	8.7195	5.7400	2.0356	3.768
3	1.5	38.384	19.192	44.411	8.1952	5.0701	2.0284	3.471
4	2	44.562	22.281	30.366	11.9854	10.8448	2.0316	5.181

Table 3 shows the structural properties of NiFeCr thin films and the film coated at high current density have lower crystallite size of 30 nm. The bath concentration is one of the important parameter which influence the particle size. For sulphate bath the current density is optimized to 2 A/dm^2 for NiFeCr thin films.

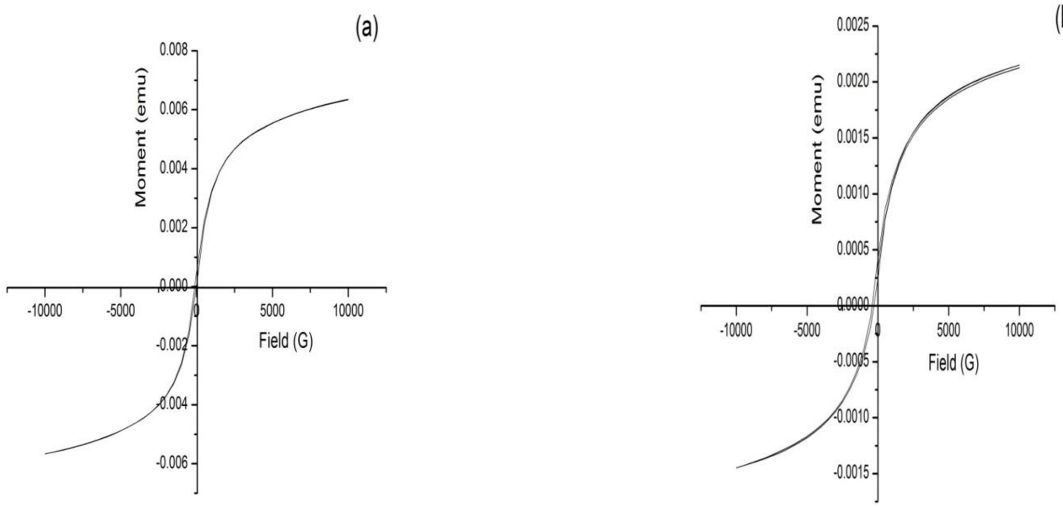
3.3 Mechanical property

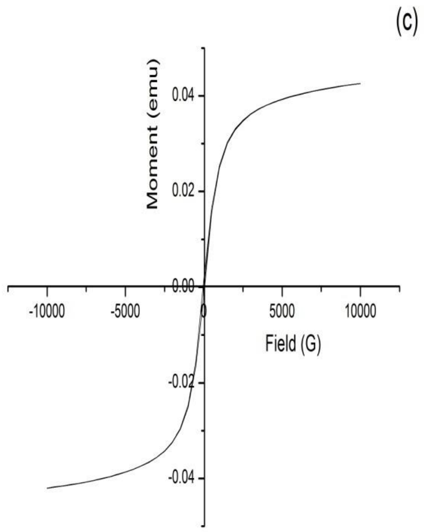
Micro hardness of electroplated NiFeCr thin films are analysed by using Vickers hardness method. All the coated films have very good adherent with substrate. This was observed by bend and scratch test. The hardness of the NiFeCr thin films are in shown in the table 4. The film coated at current density of 2 A/dm^2 have slightly higher hardness value.

Table.4 Mechanical properties of NiFeCr thinfilms

S.No	Current density	Vickers hardness(VHN)	Thickness (μm)
1	0.5	102	1.7442
2	1	115	1.3868
3	1.5	133	1.2011
4	2	159	1.6873

3.5 Magnetic Properties





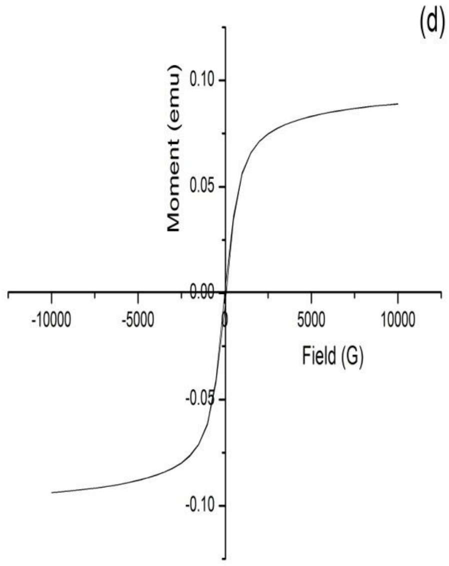


Fig.3. VSM graphs of NiFeCr films electrodeposited from sulphate bath at (a)0.5A/dm^2 (b)1A/ dm^2 (c) 1.5 A/ dm^2(d) 2A/ dm^2

The magnetic properties of NiFeCr thin films are determined by important parameters like saturation magnetisation, coercivity, retentivity and crystalline size etc. Generally the soft magnetic materials are found to have high saturation magnetisation and low coercivity. The VSM graphs for NiFeCr thin films at different current densities are shown in figure 4. The film coated from sulphate bath at 0.5 A/dm^2 has saturation magnetization of 6.0104 ×10^{-3} emu with coercivity of 55 Gauss.

If the current density increase from 0.5 A/dm^2 to 2 A/dm^2 , the saturation magnetisation increases from 6.0104 ×10^{-3} emu to 91.370×10^{-3} emu with lower coercivity of 42.8 Gauss. From VSM, we conclude that the film coated from sulphate bath at high current density have best magnetic properties compared with films coated at lower current density.

Table.5 magnetic properties of the NiFeCr thin films

S.No	Current density (A/dm^2)	Coercivity (G)	Magnetisation M_s 10^{-3} emu	Retentivity M_r Emu	Squareness S 10^{-3} (M_r/ M_s)
1	0.5	55.461	6.0104	222.98 × 10^{-6}	0.03709
2	1	62.070	1.7988	90.050 × 10^{-6}	0.05006
3	1.5	50.061	42.299	1.7289 × 10^{-3}	0.04087
4	2	42.837	91.370	3.5625 × 10^{-3}	0.03898

4. Conclusion

The structural, morphology and magnetic properties of electrodeposited NiFeCr thinfilms from sulphate bath have been studied for varying current densities from 0.5 A/dm^2 to 2 A/dm^2 . The sulphate bath current density increases the Ni content was decreased and the corresponding Fe content was increased. It is observed that the NiFeCr electrodeposits have high saturation magnetisation value with lower coercivity at high current density of 2 A/dm^2. Because of the best soft magnetic properties, the NiFeCr thin films may be used in MEMS and magnetic recording heads.

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