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Effect of Bath Temperature on Magnetic and Structural Properties of Electrodeposited NiFeCr Nano Crystalline Thin Films

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Soft magnetic thin films of NiFeCr were prepared using electrodeposition in various bath temperature. NiFeCr deposited films are textured with FCC phase preferred orientation. Experimentally observed soft magnetic property of thin films for different temperature was compared.The addition of chromium can enhance magnetic and mechanical properties of NiFe thin films. Electrodeposited NiFeCr films were prepared at different temperature (30, 50, 70 and 90 °C) and they were subjected to morphological, structural, magnetic and mechanical characterization analysis. Nickel content was maximum as 51.36 wt % at 90 °C. The chromium content increased when electrolytic bath temperature was increased. NiFeCr films were bright and uniformly coated on the surface. Also the deposits of NiFeCr films were in nano scale and the average crystalline size was around 30 nm. Thin films prepared at high temperature exhibited a high saturation magnetization and low coercivity. The micro hardness of NiFeCr was 272 VHN at 90 °C.

Keywords: Electrodeposition, Electrolytic bath, Crystalline size, VSM, Ni-Fe, X-ray diffraction, VHN, SEM.

INTRODUCTION

Soft magnetic nanocomposites are interesting materials because of their use in high frequency device application in variety of devices including on-chip micro-inductor, read write heads, magnetic sensors and spin valves [1,2]. NiFe thin films have been extensively used in practical device application [3-5]. NiFe alloy films with a composition of $Ni_{82}Fe_{18}$ is well known as a soft magnetic material and called permalloy [6,7]. NiFe films have relatively low resistivity, therefore gives rise to eddy current loss when used in microwave practical applications. Soft magnetic character of thin films are associated to crystalline nature and grain size of films [8-10]. Moreover, decrease in grain and interface role plays important role to enhance soft magnetic properties. The films should have low coercive field (H_c), high magnetization, easily achievable and controllable anisotropy with small field, high resistivity and good thermal stability in reasonable temperature range [11-13]. Electrodeposition is an electrochemical process and an old method which applied for surface structure modification. Electrodeposition method is widely used in MEMS, NEMS, communication, optical and sensors industries [14-16]. NiFeCr, NiFeW and NiW are most commonly used magnetic thin film materials in MEMS and NEMS.The electroplating of NiFeCr thin film is used to obtain enhanced soft magnetic characters, special optical properties and improved corrosion resistance [7,17,18].

EXPERIMENTAL

The electroplated NiFeCr alloy films were prepared different temperature 30, 50, 70 and 90 °C. The time duration of deposition process was 15 min. In this investigation, copper and stainless steel substrates acted as cathode and anode respectively with 1.5 cm × 7.5 cm. dimension [19]. The NiFeCr thin films were prepared from electrolytic solution which contain ferrous sulphate (10 g/L), nickel sulphate (30 g/L), chromium sulphate (15 g/L), ammonium sulphate (40 g/L), boric acid (10 g/L) and saccharin (10 g/L). The pH value of electrolytic solution was fixed as 6.0 by mixing ammonia solution and electroplating process was carried out with current density 3 mA/cm^2. The copper cathode was carefully removed from the bath after 15 min and dried for few minutes [7,19,20]. The surface nature of NiFeCr films was characterized by scanning electron microscope. The elemental content of deposits of films was examined by Energy-dispersive X-ray spectroscopy and crystal structure of deposits was analyzed by X-ray diffraction study. The micro hardness of films was measured by Vickers Hardness Test. The important magnetic properties of deposits are saturation magnetization and coercivity. Analysis of thin films with Vibrating Sample Magnetometer gives magnetic properties.

RESULTS AND DISCUSSION

Composition of NiFeCr films: Energy-dispersive X-ray analyzer (EDAX) gives elemental composition of deposits. Table-1 shows weight percentage of Fe, Ni and Cr at different electrolytic bath temperature by EDAX analysis. The films prepared at 90 °C have high content of chromium. The highest ferrous content of 26.27 wt % was found at electrolytic temperature of 30 °C. While electrolytic temperature increases, the content of nickel and chromium also increase. The higher nickel content of 51.36 wt % was found at 90 °C.

TABLE-1
EDAX ANALYSIS OF THIN FILMS

Temperature (°C)	Fe (wt %)	Ni (wt %)	Cr (wt %)
30	26.27	41.11	32.62
50	19.23	46.16	34.61
70	13.62	49.05	37.33
90	10.12	51.36	38.52

Morphological study of NiFeCr films: The surface structure of NiFeCr thin films at temperature 30, 50, 70 and 90 °C were analyzed by scanning electron microscope (SEM) images and shown in Fig. 1. The thin films are bright and uniformly coated on the surface. They are crack free by appearance.

Structural analysis of NiFeCr films: Crystal structure of NiFeCr alloy films was analyzed by powder X-ray diffraction (XRD). Fig. 2 shows diffraction patterns of NiFeCr films prepared with different temperature. The occurrence of sharp peaks in X-ray diffraction pattern exhibit crystalline nature of deposits. The XRD patterns of all samples deposited at 30, 50, 70 and 90 °C are being indicative of the (111), (200) and (220). Also XRD result reveal the existence of FCC type crystal. The particle sizes of NiFeCr deposits are 36.73, 30.89, 26.17 and 24.42 nm for the bath temperature 30, 50, 70 and 90 °C, respectively. So it is concluded that crystal size of thin film deposits decreases by increasing electrolytic temperature. Also deposits of thin films revealed nanoscale and average crystalline size is around 30 nm. When bath temperature increases, strain value increases from 0.986×10^{-3} to 1.48×10^{-3} dislocation density increases from $7.41 \times 10^{14}/m^2$ to $16.77 \times 10^{14}/m^2$.

The crystal size, strain value and dislocation density of NiFeCr alloy films are shown in Table-2. The crystalline size of deposits decreases due to onset orientation of crystals while bath temperature increases. Fig. 3 shows that the crystal size reduces with increasing bath temperature.

Mechanical properties of NiFeCr films: Micro hardness measurement of deposits was done by Vickers hardness tester. The hardness values of thin films prepared for temperature 30, 50, 70 and 90 °C are 140, 176, 214, 272 VHN respectively. So hardness test shows that the micro hardness increases by increasing electrolytic bath temperature due to lower stress

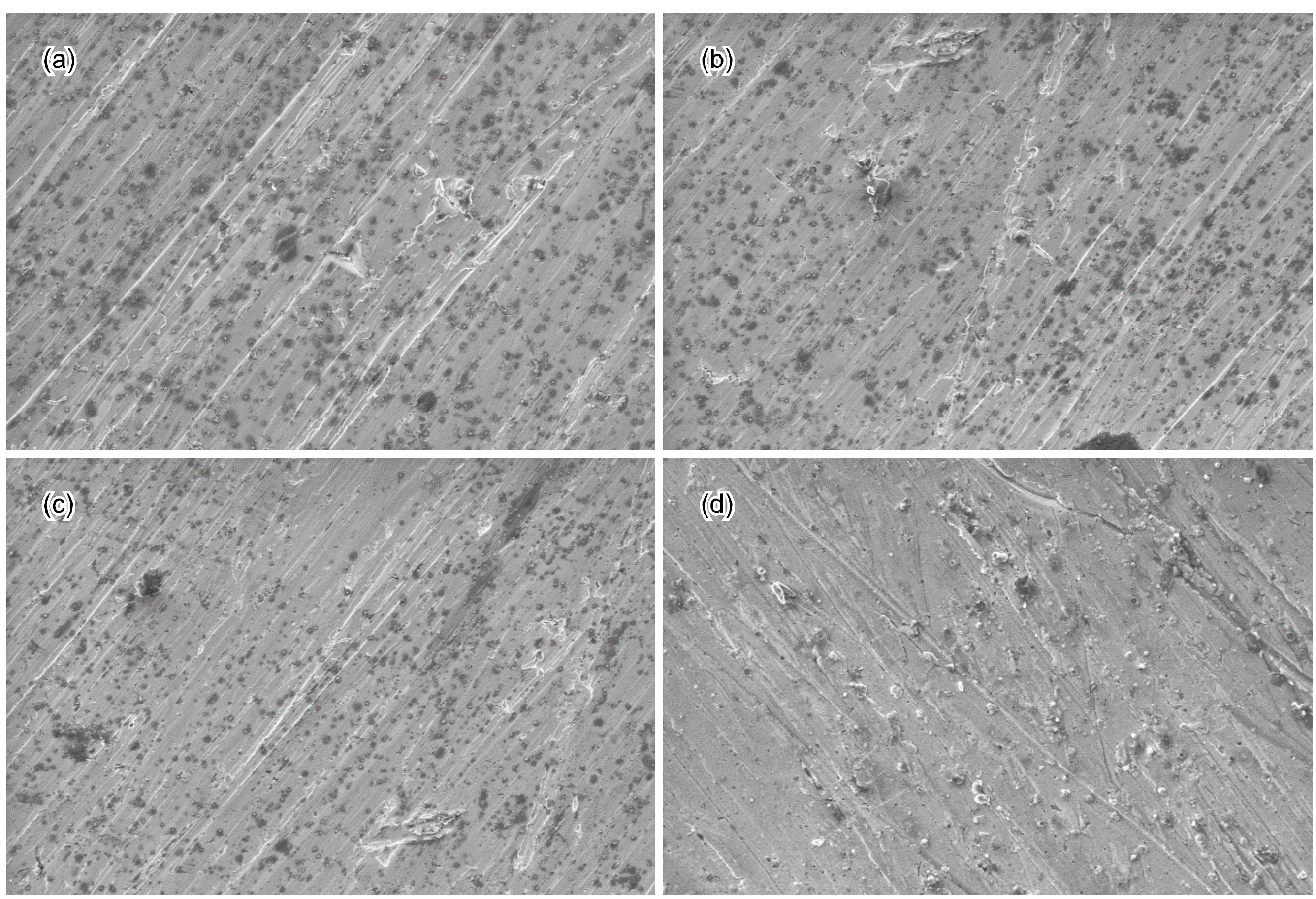

Fig. 1. NiFeCr films-SEM images at (a) 30 °C (b) 50 °C (c) 70 °C (d) 90 °C

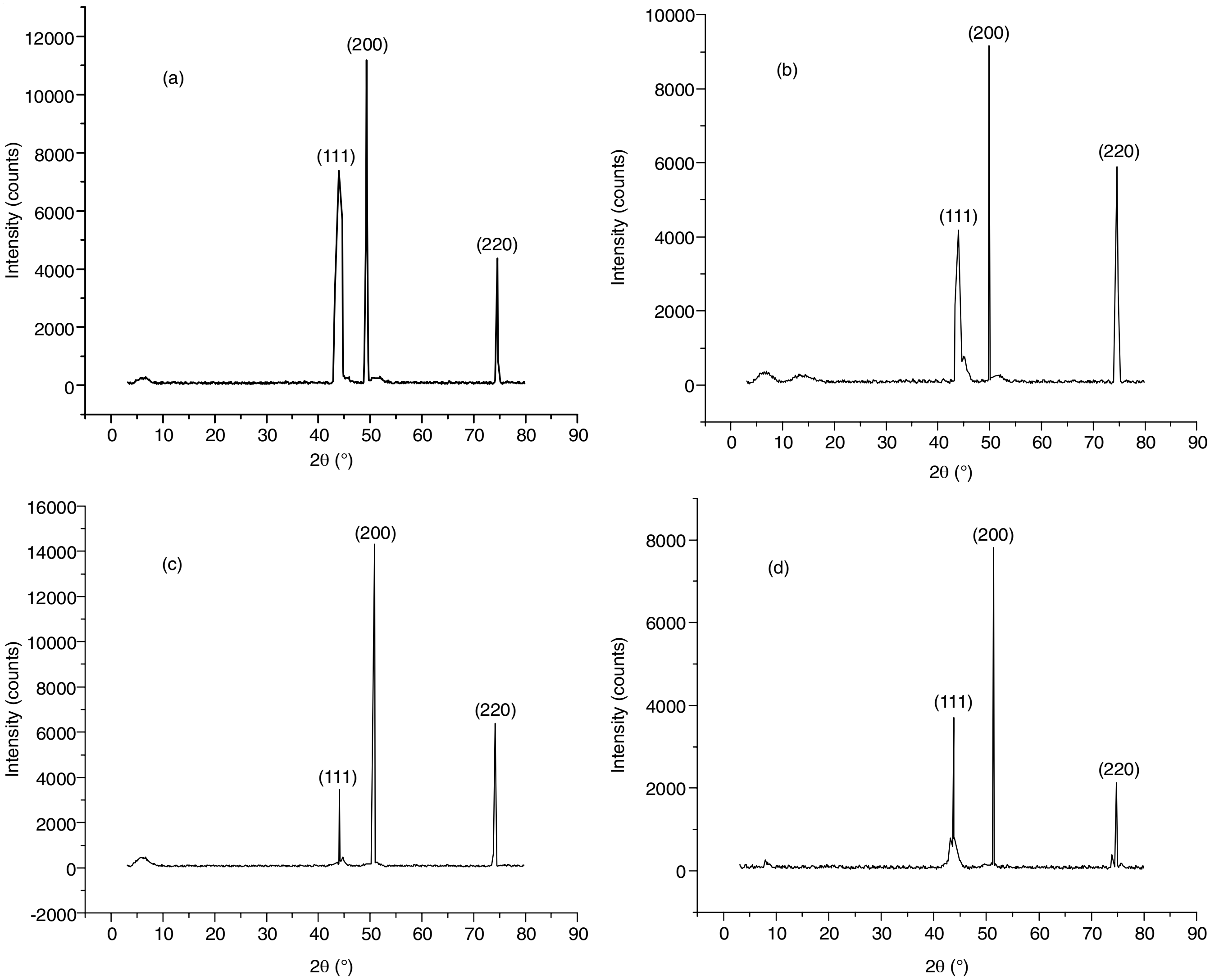

Fig. 2. NiFeCr films-XRD patterns at (a) 30 °C (b) 50 °C (c) 70 °C (d) 90 °C

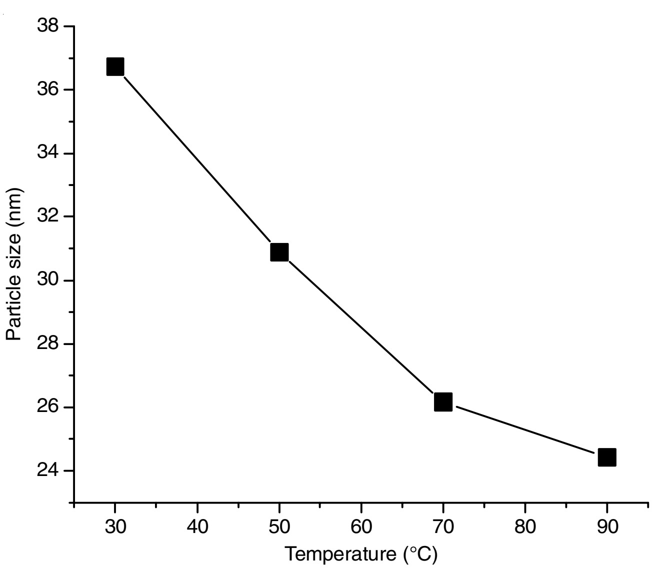

Fig. 3. Variation of crystal size with different electrolytic bath temperature

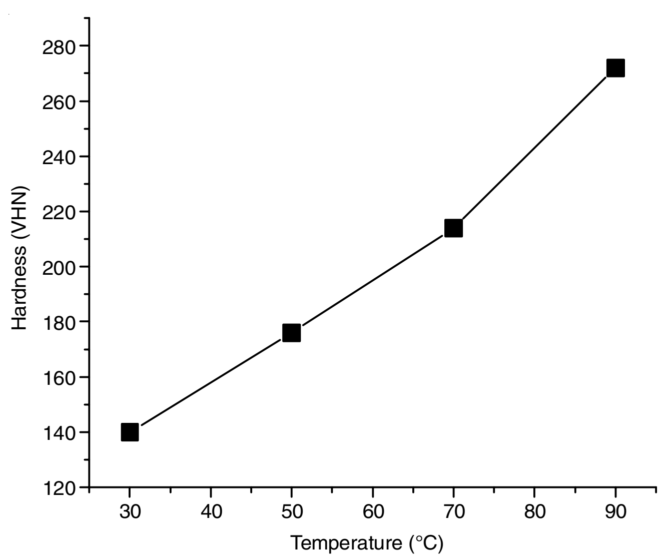

Fig. 4. Variation of micro hardness with different electrolytic bath temperature

associated with thin films. Fig. 4 shows the variation of hardness with increasing bath temperature.

Magnetic properties of NiFeCr films: The important magnetic characters coercivity, saturation magnetization, retentivity and squareness of NiFeCr alloy films were obtained

TABLE-2
STRUCTURAL CHARACTERISTICS OF NiFeCr ALLOY THIN FILMS

Bath temperature (°C)	2θ (°)	d (Å)	Particle size (D) (nm)	Strain (10^{-3})	Dislocation density ($10^{14}/m^2$)
30	49.27	1.894	36.73	0.986	07.41
50	49.83	1.915	30.89	1.172	10.48
70	50.93	1.713	26.17	1.383	14.6
90	51.38	1.961	24.42	1.480	16.77

by VSM and are given in Table-3. The hysteresis curves for thin film NiFeCr with various temperature are shown in Fig. 5. The elemental composition of deposits, applied potential, additive materials, temperature and electrolytic bath are the deciding factors of thin film magnetic properties.

When electrolytic temperature is changed as 30, 50, 70 and 90 °C, the magnetization increases from 93.72 × 10^{-3} to 222.15 × 10^{-3} emu/cm^2. Also when temperature is increased from 30 to 90 °C, the coercivity decreases from 364 G to132 G. From result of vibrating sample magnetometer, it is concluded that the films obtained at higher temperature reveals high value of magnetization and low value of coercivity. The enhanced value of saturation magnetization and reduced value coercivity promote magnetic properties of alloy coating.

TABLE-3
MAGNETIC PROPERTIES OF NiFeCr DEPOSITS

Bath temperature (°C)	Coercivity (H_s) (G)	Magnetization (M_s) (emu/cm^2) 10^{-3}	Retentivity (M_r) (emu/cm^2) 10^{-3}	Squareness (S) (M_r/m_s)
30	364	93.72	37.27	0.3976
50	245	125.83	42.51	0.3378
70	209	146.73	44.40	0.3025
90	132	222.15	51.03	0.2297

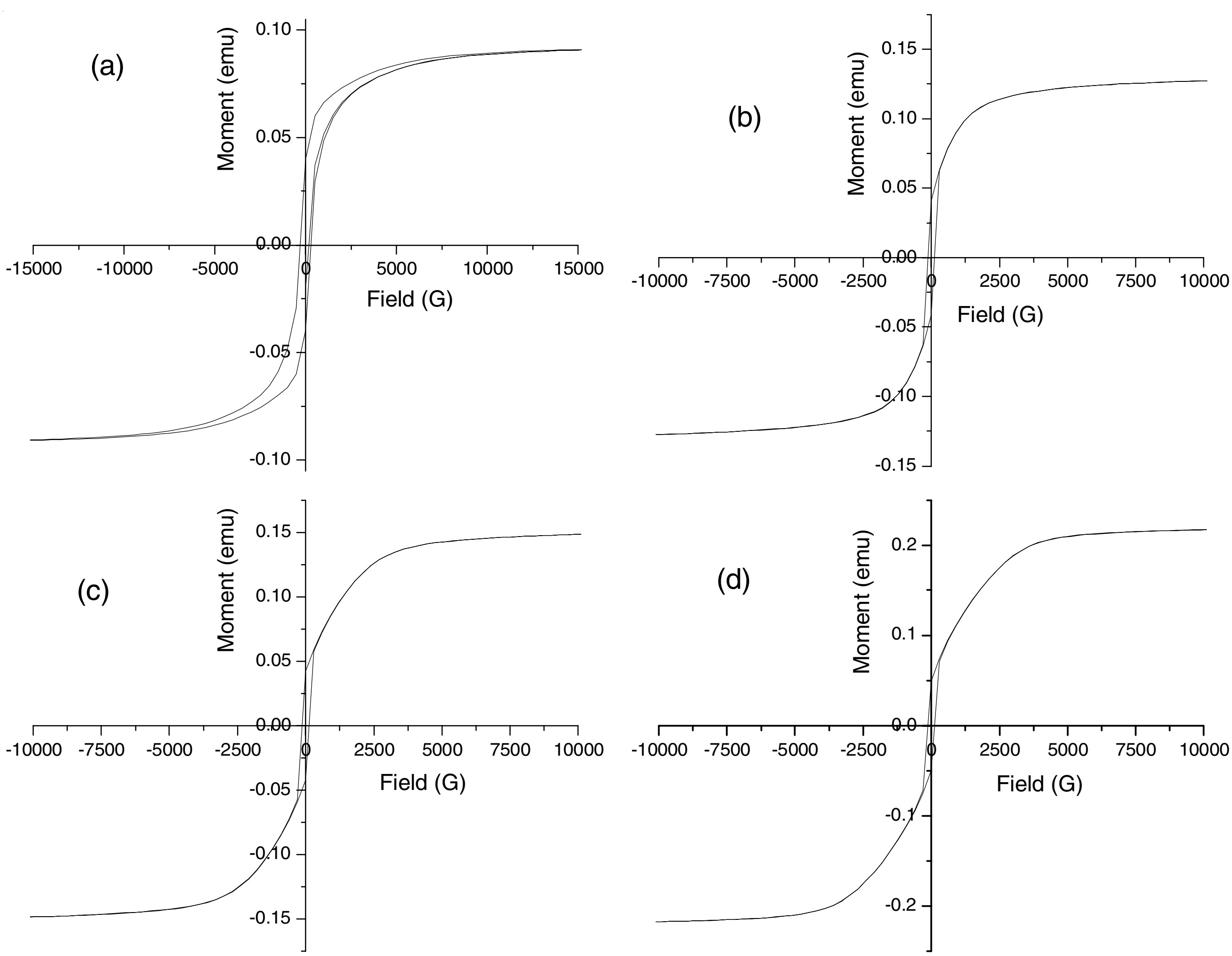

Fig. 5. Hysteresis curves for NiFeCr film at electrolytic bath temperatures (a) 30 °C (b) 50 °C (c) 70 °C (d) 90 °C

The magnetization and other important magnetic property *i.e.*, coercivity can be changed by decreasing the grain size of deposits. The film stress is reduced by increasing bath temperature. The NiFeCr thin films prepared at 90 °C have lower coercivity and higher magnetization due to low stress formation during deposition. So it is concluded that soft magnetic nature of NiFeCr thin films are enhanced by increasing bath temperature.

Conclusion

The NiFeCr alloy thin films at different temperature were prepared by electrodeposition, maintaining current density 3 mA/cm^2 and pH of solutions 6.0. The thin films are bright and uniformly coated on the surface. The XRD result reveal the existence of FCC type crystal. The crystalline size of deposits decreases due to online orientation of crystal while increasing bath temperature. The hardness increases with increasing bath temperature due to lower stress associated with thin films. When bath temperature is increased from 30 to 90 °C, the magnetization increases from 93.72×10^{-3} to 222.15×10^{-3} emu/cm^2. Also when temperature is increased from 30 to 90 ºC, the coercivity decreases from 364 G to132 G. This is due to nanocrystalline structure of deposits.The addition of chromium with NiFe alloy in electroplating can enhance its magnetic, mechanical and structural properties and this alloy films can be used in NEMS, MEMS and memory devices.

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