OPTIMISED STRUCTURAL AND MAGNETIC PROPERTIES OF NIFEP THIN FILMS BY CONTROLLING BATH TEMPERATURE FOR MEMS DEVICE APPLICATION

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Abstract

The Ni rich nano crystalline thin films of NiFeP have been successfully synthesized by electrodeposition method by varying the temperature of electrolytic solution from 30 °C to 90 °C at constant current density of 5 mA/cm2. A spectrograph of scanning electron microscope (SEM) of Energy Dispersive X-ray Analysis (EDS) were used to analyse the chemical composition and corresponding structural properties of the coated NiFeP alloy layer. The structural analysis shows that the films coated at 90 °C were bright, uniform and crack free. The EDS result indicates that the NiFeP film prepared at 90 °C has a higher Ni content of 72.14 wt % with a lower P content of 5.5 wt %. The X-ray diffraction pattern (XRD) analysis was used to confirm the nano -crystalline nature and structure of the thin film. Using a Vibrating Sample Magnetometer (VSM), the magnetic behaviour of a thin NiFeP film was analysed.The maximum saturation magnetization of 22.49 x10-3 emu with coercivity of 311 Gauss was observed for the NiFeP thin films coated at bath temperature of 90 °C. Vicker's Hardness Tester also investigated the adhesion of coated NiFeP thin films and their respective micro-hardness. Due to its improved soft magnetic nature and mechanical stability, NiFeP thin film can be used for the production of MEMS and NEMS sensors.

Keywords: *Electrodeposition; Current density; magnetic properties; NiFeP; EDS; MEMS.*

Introduction

Due to its wide range of properties, the nickels-iron alloys from the nickels to the iron-rich have a wide range of advanced technical applications. NiFe alloys have been used in industry applications for more than 100 years, due to their unusual low thermal expansion coefficient and soft magnetic properties. Magnetic alloys and possible materials have been capable of producing devices in the recent industrial world, including high density heads, micro motors, inductors, sensors, actuators and micro relays, in the Micro Electro Mechanical System (MEMS) and Nano Electro Mechanical Systems (NEMS). Magnetic alloy materials such as soft alloys are available in two forms [1-3]. Two distinct kinds of magnetic alloys, such as soft and hard magnetic alloys may be used in industrial applications. For the above applications we use mainly soft magnetic alloy materials whose nano-scale particle size is due to their increased magnetizations of saturation with higher permeability, lower coercive properties, close to zero

magnetostriction, greater resistance and lesser loss of hysteresis. The NiFe for both MEMS and NEMS devices is a well-known soft magnetic alloy [4-5]. Elements such as Co, Mn, W, Cr, etc., are applied to NiFe in order to increase corrosion resistance and magnetic nature. Among the different choices for NiFe 's third ingredient, the addition of phosphorus is expected to improve the resistance to corrosion and to wear, which in turn should improve the magnetic behaviour of the coating [6-7].

This research focused on preparation of the soft magnetic NiFeP thin film and explores its full potential in the field of MEMS devices by adjusting the temperature of an electrolytic bath temperature. Although thin NiFeP films require special care because they have a detrimental effect on the magnetic properties of films due to contamination [8-10]. The NiFe Thin film can be synthesised by various methods, such as sputtering, electron beam evaporation and electron deposition, requesting magnetic comportment improvement. The electrodeposition process is the most suitable way of producing nano-structured alloy Thin Layers based on NiFe, because of its advantages including cost-effective mass manufacturing, minimal waste of chemicals.

Experimental work

Electrodeposition of Ni rich NiFeP nano crystalline thin films were prepared with electrolytic baths consisting all precursor materials with different bath temperature. In this current research work, Nickel Sulphate, Iron Sulphate and Phosphoric acid are taken as the precursor materials for Ni, Fe and P. The experimental bath conditions for NiFeP thin films are shown in table 1. All the chemicals used for the electroplating of NiFeP were dissolved in triple distilled water to make an electrolyte. By adding the few drops of liquid NH4OH solution, the pH of the electrolyte is changed to 7.

The acid solution was created when sufficient quantities of chemicals were applied to the distilled water. For the NiFeP electrodeposition method, a copper plate with dimensions of 7.5 x 1.5 cm^2 was chosen as the substratum. Just before the deposition, the electrodes are undergone the normal cleaning process series of Emery sheet cleaning, detergent wash, sulphuric acid wash and ultrasonic cleaning, which confirms the degree of cleanliness. The required masking pattern was also issued with adhesive tape on the Cu plate. The galvanostatic potential was applied to prepare NiFeP nano crystalline thin film over the surface of copper plate. The bath temperature was varied from 30 °C to 90 °C in the interval of 20 °C in this electrodeposition process for 30 minutes. The NiFeP coated copper plate was rinsed with triple distilled water for further characterisation analysis after deposition. All prepared NiFeP nanocrystalline thin films were subjected to several characterization testing like SEM – EDS, XRD, Vickers hardness and VSM. Further, the respective results of analysis were also discussed.

Table 1. Electrodeposition bath details of NiFeP alloy films.

S. No.	Name of the chemicals	Required values (g/lit)
1.	Nickel sulphate	40
2.	Ferrous sulphate	30
3.	Phosphoric acid	15
4.	Boric acid	10
5.	Citric acid	10

Results and Discussions

Structural analysis– SEM – EDS

The SEM spectrum is currently collected by using Hitachi S-3400 electron scanning microscope. The dispersive energy spectra are recorded using a TESCAN VEGA LMU3 scanning electron microscope in conjunction with the Bruker EDS spectrometer. In EDS spectra, the predominant peaks match to the presence of chemical elements like Ni, P and Fe in the prepared samples. Table 2 shows the percentages of atomic weight of the individual components in the prepared samples. The EDX chemical composition analysis shows that the Ni to Fe atomic ratio is about 2:1 and thus shows that NiFeP is prepared stoichiometrically well. Figures 1 & 2 show SEM and EDX spectrums of the samples of nickel ferrite. The SEM images show NiFeP nano particles being aggregated under both as prepared conditions and different conditions in the electrolyte bath that reveal the strong dipole attractions between the particles.

Table 2. EDS analysis of NiFeP thin films

S.No	Element	Atomic (%) Sample - A	Sample - B	Sample - C	Sample – D
1	Ni	65.51	70.64	67.46	72.14
2	Fe	28.42	24.06	26.16	22.36
3	P	6.07	5.30	6.38	5.50

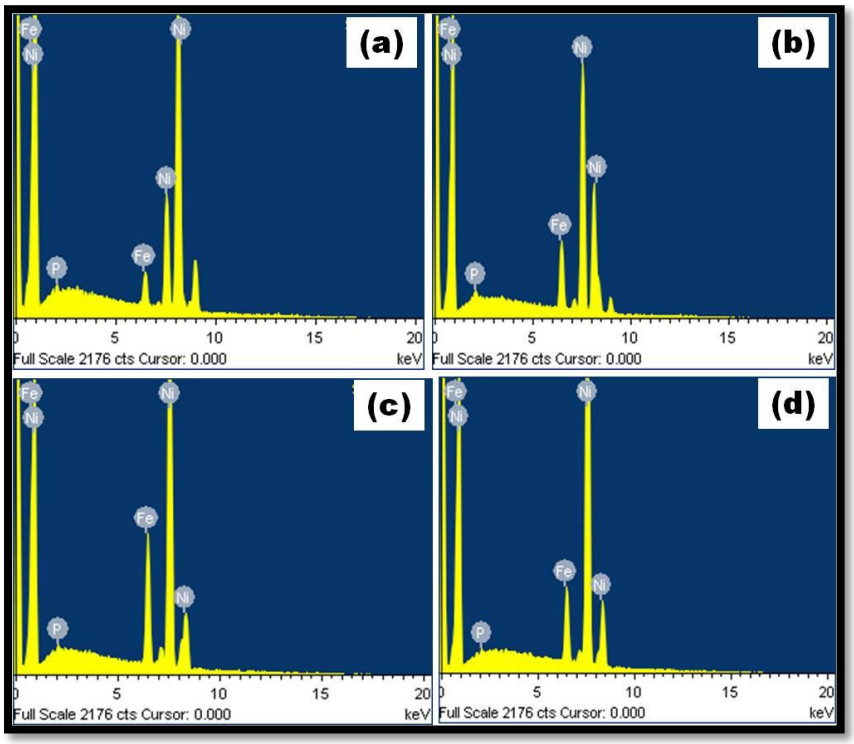

Fig 1 EDX spectrum of NiFeP thin films deposited for bath temperature of (a) 30ºC, (b) 50ºC, (c) 70ºC (d) 90ºC

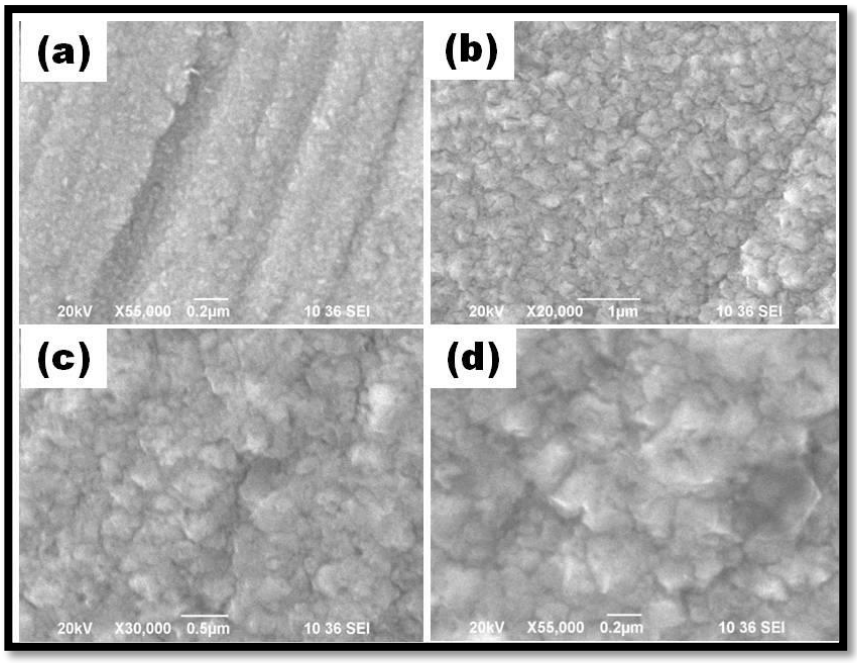

Fig 2 SEM pictures of NiFeP thin films deposited for bath temperature of (a) 30ºC, (b) 50ºC, (c) 70ºC (d) 90ºC

XRD Analysis

The graphs of X-ray diffraction for NiFeP alloy films processed in copper substrates for various bath temperatures are illustrated in Figure 3. The peaks of diffraction observed are corresponding in all cases to NiFeP's tetragonal structure. Furthermore, there can only be no diffraction peaks from Ni which reveal NiFeP single phase formed. The XRD peaks are seen as declining with a rise in the temperature of the bath. With a rise in temperature, the intensity of reflection increases. All films show the random orientation of crystalline nature in the material from this analysis. Increased crystallinity results in higher peak intensities of an XRD pattern and the cluster of particles can be attributed to higher crystallite sizes. The maximum intensity of (112) reflections was observed and thus confirms the NiFeP films preferred orientation is (112) plane. For the tetragonal structure, the film's lattice parameters are determined based on observations of 2θ and d-values. Table 3 lists the values obtained from the XRD data. As in the Scherrer equation, the W-H technique does not follow a dependency of $1/\cos\theta$ but rather varies with $\tan\theta$ alternatively. This basic difference allows a reflection separation to expand when a small crystallite and micro strain occurs together. A plot is drawn from $4\sin\theta$ to $\beta\cos\theta$ in the case of NiFeP films. The strain (μ) is estimated from the slope value of the linear adaptation to the data, and values are indicated in table 4.

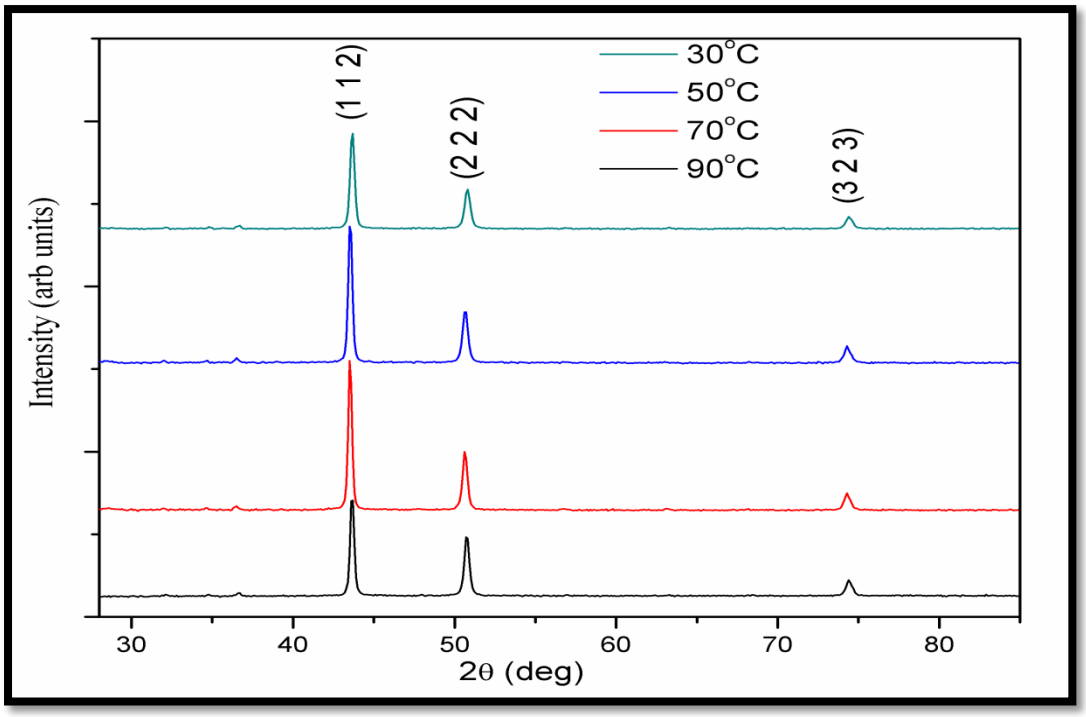

Fig 3 XRD patterns of NiFeP thin films

Table 3. The microstructural parameters of NiFeP thin films

S.No	Bath temperature (°C)	d- value (Å)	Thickness t (nm)	Cell parameters (Å) a = b	C	c/a
1	30	2.2583	877	3.2893	5.2854	1.6068
2	50	2.2678	916	3.2244	5.2356	1.6237
3	70	2.2693	946	3.2945	5.3063	1.6107
4	90	2.3247	965	3.3036	5.2844	1.5996

Table 4. Geometric parameters of the NiFeP thin films using different bath temperature

S.No	Bath temperature (°C)	Crystalline size D (nm)	Lattice strain ε (10-3) no unit	Linear stress σ (GPa)	Dislocation density δ (1012) m-2
1	30	41.7909	27.4265	356.36	57.25
2	50	46.6373	21.9038	287.70	45.97
3	70	51.1818	16.5908	215.67	38.17
4	90	68.5339	8.3362	108.29	21.29

Mechanical properties

Hardness tests are used to determine the mechanical strength of the materials and they correspond to other mechanical characteristics such as elastic constants and stress. The resistance of a material to plastic deformation due to scratching or indentation, which is called material hardness. Vickers Hv of the material has been calculated with $Hv = 1.8544\ P/d^2$ Pascal in which P is the load applied and d is the average diagonal length of the indented impressions in metres. Micro-hardness studies have been performed on NiFeP film with an incident light

microscope-mounted Leitz Vickers micro-hardness tester fitted with a Vickers diamond pyramid indenter. Adhesion of the deposited films with the substrate approves the excellent adhesion of the Cu substrate to the NiFeP alloy that is deposited at different bath temperatures. Table 5 shows the hardness values of the films that are deposited at various bath temperatures. With increased bath temperatures from 30oC to 90oC, the results showed that hardness slightly decreases. Due to their lower stress of electrodeposited NiFeP thin films, the film hardness decreases from 364 VHN to 219 VHN.

Table 5. Mechanical properties of electrodeposited NiFeP thin films

S. No	**Bath Temperature °C**	**Vicker's Hardness VHN**
1	30	364
2	50	256
3	70	237
4	90	219

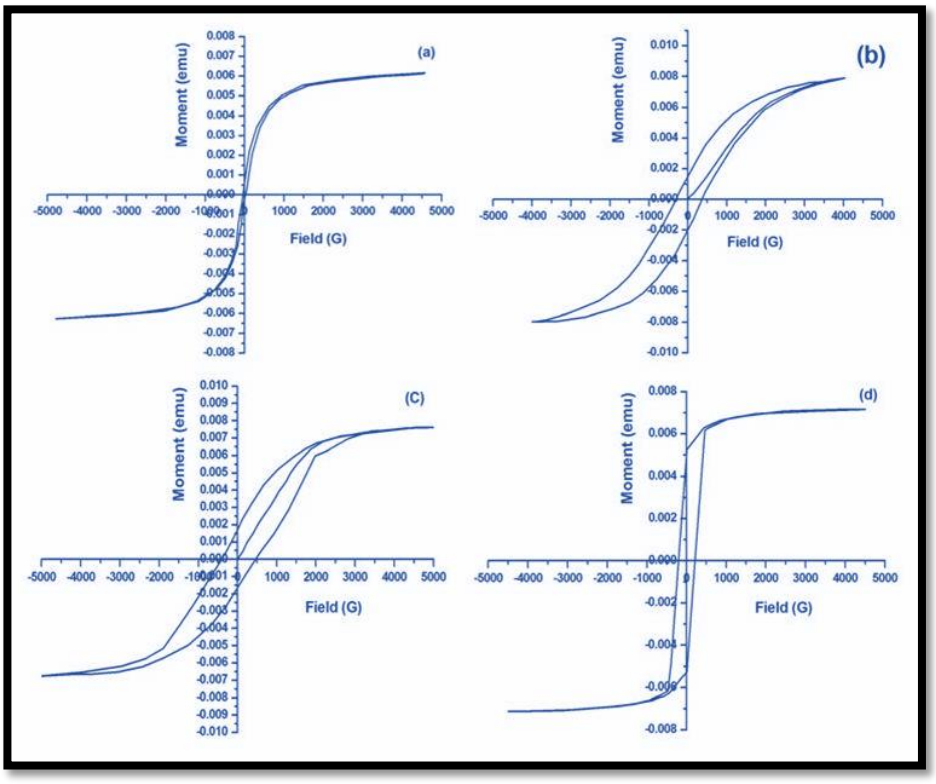

Fig 4 Hysteresis loops of Ni-Fe-P alloy thin films at different bath temperature (a) 30°C (b) 50°C (c) 70°C (d) 90°C

Magnetic properties

The thin films deposited with various bath temperatures (30 ° C, 50 ° C, 70 ° C and 90 ° C) of the NiFeP-alloy were subjected to vibrating sample magnetometer to study the The important magnetic parameters. The room temperature magnetic hysteresis loops are shown in Figure 4. It is shown that the plots of hysteresis are well defined magnetic loop and have soft magnetic features. Magnetic parameter values of NiFeP thin films are derived from hysteresis loops and given in Table 6. It is obvious that the bath temperature and coercive nature of the films deposited have a linear relationship. This is because that the decreasing coercive (Hc) value is observed with increasing bath temperature from 30 ° C to 90oC. The value of magnetic saturation value increases steadily with an increase in the bath temperature value from 30 to 90oC. This is due to the decrease in coercion (Hc) value. The retentivity value increases while the bath temperature rises and the maximum value at 90oC is 394.23 $x10^{-6}$. But the squareness variation from 30^{o}C to 50^{o}C is insignificant due to the rise of the bath temperature. But, beyond the bath temperature 50^{o}C, the variation of squareness with bath temperature decreases and attain the minimum value (17.52) at high bath temperature (90^{o}C). Thus, at high bath temperature the film achieved the desirable low Hc and high Ms values.

Table 6. Magnetic properties of the NiFeP alloy thin film at various electrolytic bath temperature

Bath Temperature Density (^{o}C)	Coercivity, Hc (G)	Saturation Magnetisation, Ms ($x10^{-3}$ emu)	Retentivity, Mr ($x10^{-6}$ emu)	Squareness, Mr/Ms (10^{-3} emu)
30	330.52	6.296	299.51	47.57
50	332.54	13.537	308.23	22.76
70	367.36	17.562	327.75	18.66
90	311.73	22.493	394.23	17.52

Conclusions

The NiFeP Nanocrystalline thin films have successfully been synthesised over Cu substrate for 30 minutes period by electrodeposition at various bath temperatures. The EDS spectrum of prepared thin films disclose the presence of Ni, Fe and P components with a high atomic percentage of Ni. The SEM micrograms clearly show that the NiFeP thin films were crack free, uniform and bright with spherical shaped crystallites. The XRD analysis concluded that the prepared thin films have a tetragonal crystal structure with a smallest particle size of approximately 41 at room temperature. With the lowest coercive and higher magnetic saturation, the NiFeP thin films exhibited soft magnetic nature. The hardness of the film decreases from 364 VHN to 219 VHN while increasing the bath temperature. The soft magnet and mechanical properties observed allow for the production of MEMS / NEMS-devices including micro sensors with the prepared NiFeP Thin Alloy films.

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