Synthesis, Structural and Mechanical Properties of Electroplated Nifemo Nanocrystalline Thin Films

R.Kannan, M.Selvambikai, E.Selva kumar, S. Venkateshwaran

Abstract— ***This study focuses on nano crystalline thin film coating of NiFeMo on the surface of mild steel at three different bath temperatures such as 60°C, 70°C and 80°C by elctrodeposition technique. Surface analysis and elemental compositional analysis of coated thin films have been employed with the help of SEM images, EDAX and XRD patterns. Electrochemical technique has been used to investigate corrosion behaviour on electroplated NiFeMo thin films. SEM analysis confirms that the existence of bright and spherical shaped granules and crack free surface on the coated film. The particle size of coated thin film was calculated using Scherrer formula and it was in the range of few nanometres. XRD study confirms that the film coated at elevated bath temperature has tetragonal structure and the remaining have mixed phase of cubic and HCP structures. Electrochemical studies discloses that all the NiFeMo thin films coated at three different temperatures have better corrosion resistance as compared with NiFe and NiFeW thin films. Among the three different bath temperatures the film coated at 60◦C has better corrosion resistance as 150.17Ω. Based on the obtained structural and corrosion results of the synthesised thin films, they are highly suitable for manufacturing of MEMs and NEMs devices***

Keywords: Corrosion resistance, electrodeposition, molybdenum, MEMS and NEMS

I. INTRODUCTION

Now a day's very powerful sensors and actuators are promisingly produced by means of MEMs and NEMs technology. Miniaturization, optimization and cost reduction are the major advancements in MEMs and NEMs based electronic devices but they have been limited by the electrical, magnetic and mechanical properties of thin magnetic alloy coatings. There are wide variety of magnetic alloys that can be plated for MEMs and NEMs, among those NiFe is the popular alloy because of its low internal stress, low magnetostriction, good mechanical properties, high saturation magnetization and high permeability [1-4]. But controlling corrosion behavior and enhancing magnetic properties of NiFe are still a challenge. In the recent days researchers looking for suitable third metal element to enrich the corrosion resistance and magnetic properties of NiFe [5-8]. Generally refractory metal elements such as tungsten, molybdenum, niobium, tantalum etc., have very good mechanical properties such as high tensile strength, highest young's modulus of elasticity, and high corrosion resistance, excellent thermal properties like high melting point and low coefficient of linear thermal expansion. In this study, the refractory metal element Molybdenum (Mo) which attributesmelting point of 2620◦C, thermal expansion of 4.8 μm/m.K, young's modulus of 329 GPa, Vickers hardness of 1400-2740 GPa, was chosen as a third element and NiFeMo was coated on the surface of mild steelplate. Amid various coating techniques electroplating method was employed because the properties of coated thin film on the substrate can easily be controlled by varying electrolyte concentration and deposition parameters [9-12]. In this research work NiFeMo alloy was successfully electroplated on the surface of mild steel substrate at different bath temperatures such as 60°C,70°C and 80°C.

II. EXPERIMENT

The NiFeMo alloy was coated as thin film on mild steel substrate by electro deposition method at three different bath temperaturessuch as 60°C,70°C and 80°C. Electrolyte bath was prepared by dissolving all the required reagent graded chemicals in triple distilled water. Few drops of ammonia solution was addedin the bath with the intension to adjust the PH value to 8. Table 1 shows the chemical composition and optimized bath conditions. Mild steel plate of 7.5 cm length and 1.5 cm breadth was used as cathode and the same dimension of pure stainless steel plate was used as an anode. Initially both the plates were well cleaned with soap water and further dipped in sulphuric acid for fine cleaning and finally rinsed with triple distilled water. Adhesion tap was used to mask off the surface area which are not desired for the thin film coating. The NiFeMo alloy was electrodeposited at three different temperatures such as 60°C,70°C and 80°C by applying constant current density of 1 A/dm^2. Several characterization techniques like SEM , EDAX ,XRD and electrochemical studies have been employed for all the coated thin films and the results are presented here.

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Table1. Optimizationof bath conditions forNiFeMo.

S.No	Name of the chemical parameters	Data
1	Nickel sulphate	60 g/l
2	Ferrous sulphate	30 g/l
3	Sodium molybadate	10 g/l
4	Diammonium citrate	60 g/l
5	Citric acid	5.5 g/l
6	Boric acid	10 g/l
7	pH value	8
8	Temperature	60, 70 and 80 (˚C)
9	Current density	1 A/dm2

III. RESULT AND DISCUSSION

EDAX analysis

EDAX analysis confirms that the deposition of nickel, iron and molybdenum particles on the surface of the mild steel plate. The EDAX spectrum indicates that the 45% of Ni, 25% of Mo and 15% of Feare coated on the mild steel surface. Figure 1 shows the EDAX spectrum of synthesized thin films.

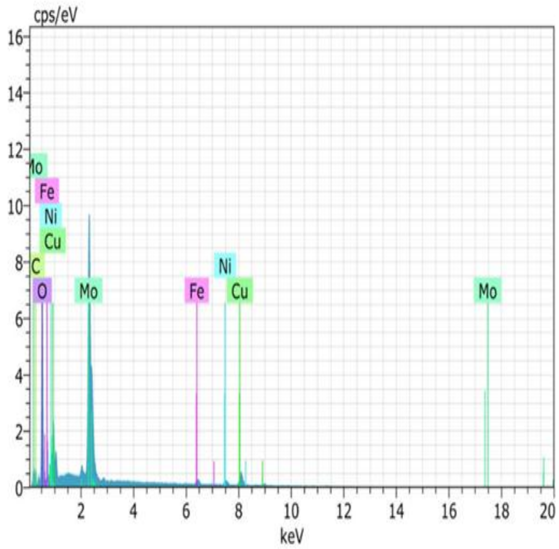


Figure 1 EDAX spectrum of NiFeMo

SEM image analysis

The micro structure and surface analysis of coated thin film samples have been analyzed from SEM images. SEM image of the NiFeMo coated area of the mild steel substrate reveals that the existence of bright and spherical shaped granules. SEM micrograph confirms that the surfaces of the coated thin films are crack free and the shape of the nano crystalline molecules changes from spherical to needle shape as the temperature increases to higher range. Figure2 shows the micro structures of the NiFeMo thin film coating.

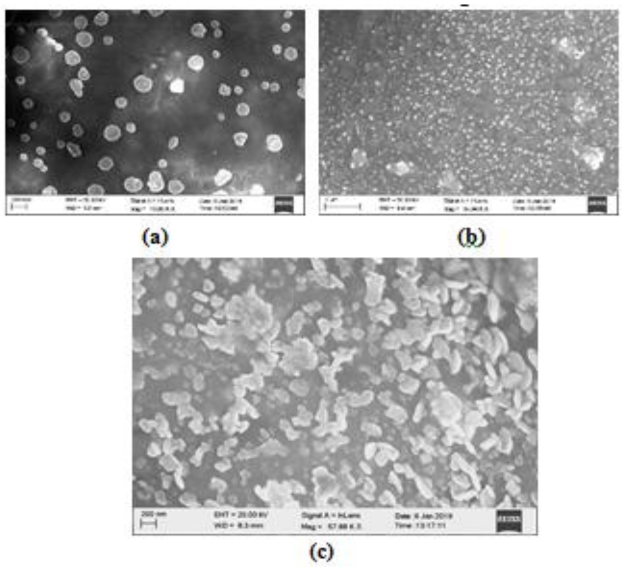


Figure 2. SEM images of NiFeMo thin films (a) coated at 60°C (b) coated at 70°C (c) coated at 80°C

The NiFeMo thin film coated at bath temperature 80°C exhibit the needle shape and mushroom shaped particles as shown figure 2.SEM micrographs reveal that the particle diameter is decreased from 185.4 nm to 118.4 nm by increasing the bath temperature from 60°C to 80°C and pictures are shown in figure 3.

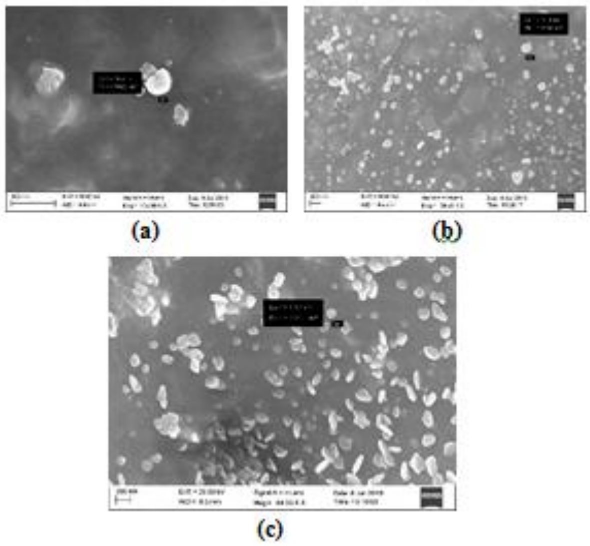


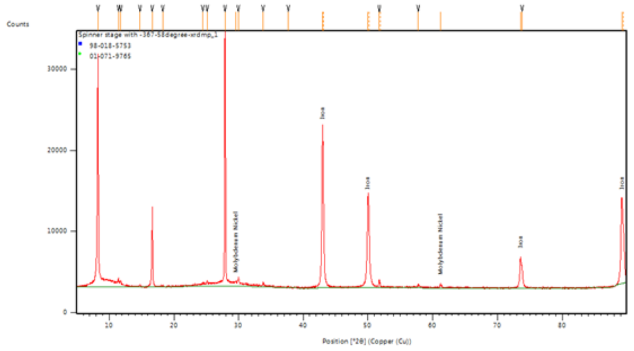
Figure 3. Particle size of NiFeMo thin films (a) coated at 60°C (b) coated at 70°C (c) coated at 80°C

X Ray diffraction study

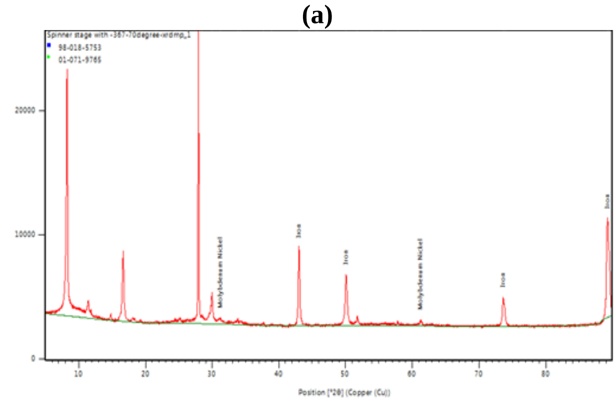
All the synthesized NiFeMo thin films were subjected to structural analysis and the obtained graphs are shown in figure 4. X ray diffraction study has been used to calculate the particle size and crystalline nature of the nano crystals existing in the coated NiFeMo thin film. Particle size has been calculated from Scherrer's formula and it was found that the particle size is changing with respect to bath temperature, ranging between 25-30 nm. The film coated at 80◦C has the particle size of 25nm. This is due to the reorientation of atoms during the time of electroplating.The coated thin film exposes perfect cubic structure at higher bath temperature of 80◦C and it has mixed phase of cubic and tetragonalstructures at 60◦C and 70◦C. Fig 3 shows the Xray diffraction pattern of NiFeMo thin films. The results obtained from XRD are presented in table 2.

Table 2.XRD results of NiFeMo thin films

Bath temperature	**2 θ (deg)**	**Lattice parameter a (A^0)**	**Crystallite size D Nm**	**Strain 10^{-4}**	**Dislocation density (10^{14} / m^2)**
60	28.20	9.4219	26.67	13.58	14.0590
70	40	9.4219	31.40	11.53	10.1424
80	80.10	9.4414	25.25	14.34	15.6847



(a)



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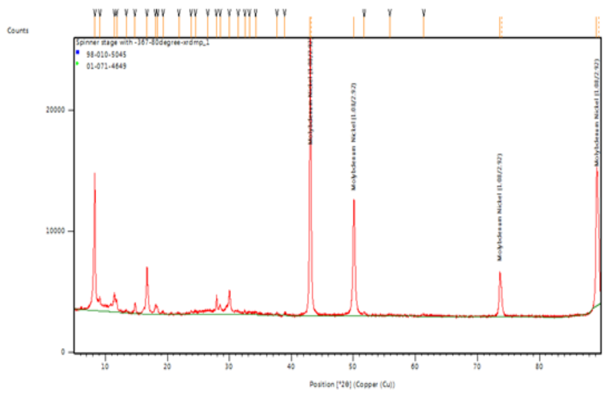


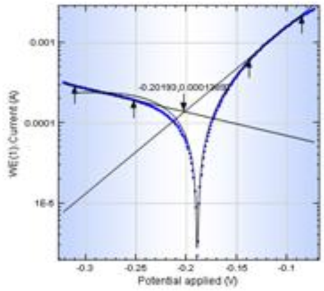
(c)

Figure 4. XRD patterns of NiFeMo thin films (a) coated at 60°C (b) coated at 70°C (c) coated at 80°C

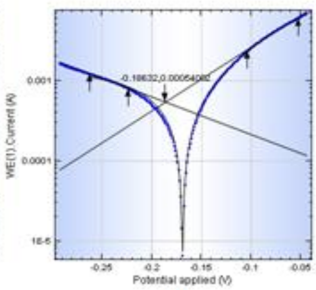
From XRD data, it is concluded that the NiFeMo thin films coated at higher bath temperature have approached to perfect nano level and the phase transition also occurs from mixed phase of cubic and tetragonal to perfect cubic structure.

Corrosion Studies

Corrosion behavior of NiFeMo coated thin films was analyzed using electrochemical studies such as electrochemical impedance spectroscopy and potentiodynamic polarization.The polarization parameters such as corrosion potential (**Ecorr),** corrosion current densities**(icorr)** and polarization resistance were tabulated in Table 3 and the corresponding Tafel plot is shown in fig 5. The tabulated results which are obtained from electrochemical studies disclose that the corrosion current drastically decreases, corrosion rate tremendously falls and the corrosion resistance extremely higher at the lower temperature compared with the elevated temperatures.



(a)



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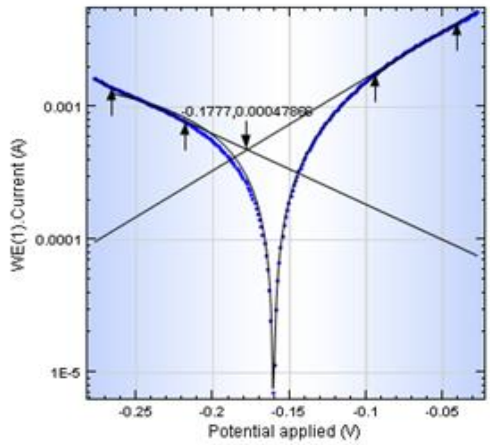

(c)

Figure 5. Polarization study of NiFeMo thin films (a) coated at 60°C (b) coated at 70°C (c) coated at 80°C

Impedance analysis result reveals that the charge transfer resistance of the NiFeMo thin film electroplated at 60◦C is higher than the films coated at 70◦C and 80◦C. The extent of corrosion resistance is improved to 40.68% for the film coated at higher temperature.

Table 2. Corrosion results of NiFeMo thin films

Test Samples	**Ecorr, (V)**	**ba (V/dec)**	**bc (V/dec) (V/dec)**	**icorr (A)**	**Polarization resistance (Ω)**
NiFeMocoated at 60**°C**	-0.20193	-0.44896	0.1424	0.000603	150.17
NiFeMocoated at 70**°C**	-0.18632	-0.93345	0.20018	0.002453	45.122
NiFeMocoated at 80**°C**	-0.1777	1.6901	0.21321	0.001526	53.871

IV. CONCLUSION

The NiFeMo alloy is successfully coated on the miled steel substrate using electrodeposition technique at three different temperatures. The surfaces of all the coated thin films are crack free. The particle size of thin films are varying with respect to the bath temperature and the particle size is reduced to 25nm for the film coated at 80◦C. The corrosion resistance of the film coated at 60◦C is tremendously increased as compared with films coated at elevated temperatures.

V. ACKNOWLEDGEMENT

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