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Effect of the Additive on the Properties of Ni-Fe Coating

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Electrodeposited Ni-Fe coating is employed in many fields such as corrosion, wear, magnetic and electrical applications, and electrocatalytic materials. To improve the properties of Ni-Fe coating, a wide variety of properties for coatings can be achieved by selecting different electrodeposition parameters. In this work, Ni-Fe coatings are deposited on copper substrates. X-ray diffraction (XRD), scanning electron microscope (SEM), energy dispersive spectroscopy (EDS) and microhardness analysis are used to study the effects of boric acid on the morphology, phase structure, nickel content and microhardness of the coatings. The results confirm that the XRD patterns of the alloy deposits reveal that there are a number of sharp peaks in the images, indicating good crystallinity. The morphology of electrodeposited Ni-Fe coatings shows that the grains are spherical in nature for all samples, and EDS analysis of Ni-Fe composite confirms that the reduction of Ni decreases with increasing boric acid concentration. Moreover, reducing the concentration of boric acid in the Ni-Fe coating bath can lead to improved properties.

Keywords: Ni-Fe coating, Morphology, Boric acid, XRD.

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1. INTRODUCTION

Ni-Fe coating is used in the electronics industry, mainly in various fields of specific technological applications, such as magnetic devices in electrical component boxes, space telescope armatures, machine tool parts, for example, to reduce production costs and to provide soft magnetic properties, and as a memory device, exhibiting excellent properties, such as low thermal expansion coefficient, low coercivity, high saturation magnetization, relatively high permeability, magnetic and corrosion-resistant properties and special optical properties, and it is currently a key alloy for many applications due to its very low coefficient of thermal expansion [1-6]. To obtain coatings on substrates, various methods, with different degrees of success, have been used for this matter, such as molecular beam epitaxy, sputtering, evaporation, electrodeposition and electrolysis. Electrodeposition is one of the most used methods for coating deposition over metallic substrates due to its simplicity, uniform and controlled deposition rate, operation at room temperature, flexibility, the ability to obtain multilayers and to coat large surfaces in various forms at low cost [1-3, 7-10]. Ni-Fe coatings were dictated by a number of factors, including the metallurgical structure and alloy composition. These parameters were affected by processing variables such as bath composition, pH, current density, temperature and boric acid (additives) [11, 12]. Several studies have investigated the effects of additives in the bath [13, 14]. The addition of saccharin to the solution reduces the iron and nickel deposition rates; on the other hand, boric acid acts as a surfactant, blocking the reduction of nickel but allowing the reduction of iron [13]. In another study, the presence of ethylenediamine in chloride baths considerably increased the nickel content in the deposits [15], but unlike additives, such as citric acid, formed complexes with iron and nickel ions, and the addition of additives like citrate, tartrate, gluconic, tartaric, citric and glucose acids, in the bath increased the iron content [12]. In this study, the influence of boric acid concentration on the composition, morphology, phase structure, and microhardness of the alloy deposits are investigated.

2. EXPERIMENTAL

2.1 Preparation of Ni-Fe Coating

The substrate used in this study is a copper sheet with an area of 10 × 20 mm². The substrate was polished with 120 to 4000 grade SiC paper and then rinsed with distilled water, degreased in 50 g/l Na_2CO_3 and 15 g/l NaOH solutions, then pickled in 10 % HCl solution to remove oxide traces, and finally washed with distilled water. The experimental setup for electrodeposition of Ni-Fe coating is shown in Fig. 1. The substrate (Cu) is placed in the middle of two anode (Ni) plates to achieve double-sided plating growth on the substrate. In addition, it is necessary to ensure that two anode plates are parallel to the cathode substrate and the distance is kept at 1 cm to ensure the same plating quality on both sides of the plated part. Composition and parameters of composite plating bath is shown in Table 1.

2.2 Deposits Characterization

To test the coatings adhesion, the samples were heated for 30 min at 250 °C and then immersed in water at room temperature [16]. The surface morphology and elemental composition of the coatings were studied by environmental scanning electron microscopy (FEI QUANTA 200) coupled with energy dispersive X-ray diffraction microanalysis (EDX), which was also used to examine the weight percent content in the composite coating.

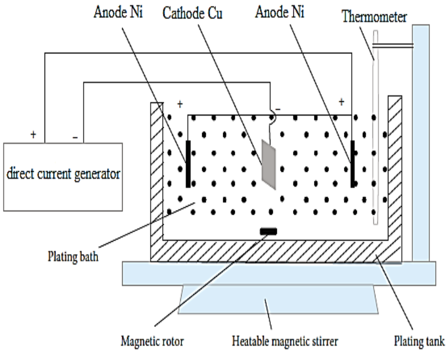


Fig. 1 – Experimental setup for electrodeposition of Ni-Fe coatings

Table 1 – Electrodeposition bath chemical composition and working conditions

Bath composition	Concentration (g/l)	Conditions
$NiSO_4$, $7H_2O$	9.46	Room temperature
$(NH_4)_2$ $Fe(SO_4)_2$, $6H_2O$	25.09	Current density 1 A/dm^2
NaCl	5	pH 5-6
$C_7H_5NO_3S$	0.5	Deposition time 10 min
$NaC_{12}H_{25}SO_4$	0.4	–
H_3BO_3	0,6,10,12	–

The crystalline phases and preferred growth orientation of the coatings were evaluated by X-ray diffraction (XRD, Mini Flex 600, Rigak). Data from $2\theta = 10$-900 were measured with a step size of 0.040 (2θ). The mean crystallite size was calculated using the Debye-Scherrer equation [16]:

$$D = k\lambda/(\beta \cos\theta),$$

where k is the Scherrer constant; λ is the wavelength of X-ray (CuKα radiation, $\lambda = 1.54059$ Å); β is the FWHM, and θ is the Bragg angle. The microhardness of the deposits was measured by a Vickers microhardness tester (Wilson 402UD Wolpert), with an applied load of 100 g for 10 s. The reported values are the average of five measurements.

3. RESULTS AND DISCUSSION

3.1 Effect of H_3BO_3 on the Deposits Structures

Fig. 2 shows the XRD patterns for Ni-Fe coatings prepared at different concentrations of boric acid. As seen, the patterns present four main peaks assigned to $Fe_{0.94}Ni_{0.06}$, Fe-Ni, Ni and Cu phases, namely diffraction peaks at 44.59° for Ni, at 50.47° for NiFe, and 64.87° and 82.30° for $Fe_{0.94}Ni_{0.06}$. The crystallographic planes of the peaks assigned to phases are (111), (000), (200) and (211), respectively. In addition, the relationships between the grain sizes, Ni and Fe contents and boric acid concentration of Ni-Fe coatings are presented in Fig. 2. This indicated that the grain sizes of deposits increased from 22 nm to approximately 25 nm firstly as Ni content increased from approximately 15.43 wt. % to approximately 36.18 wt. % (Table 2).

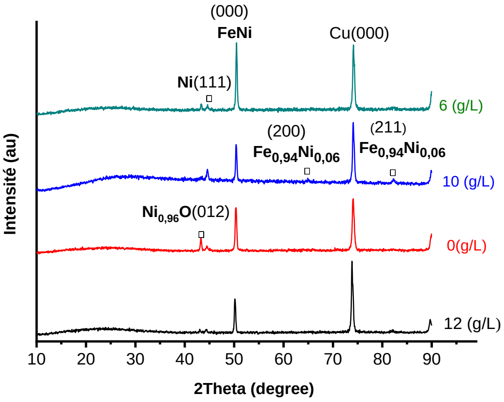


Fig. 2 – XRD spectra of Ni-Fe coatings as a function of different boric acid concentrations

Table 2 – Effect of boric acid concentration on grain size and Ni, Fe content in the coatings

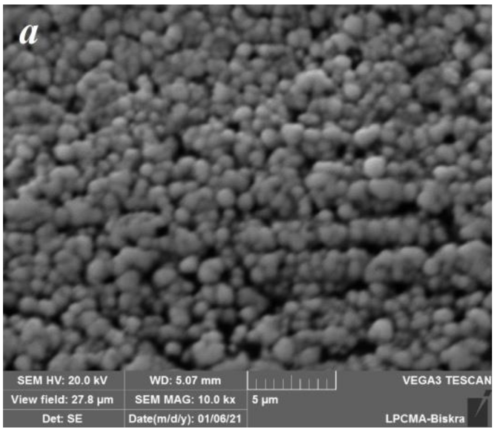
Concentration (g/l)	Grain size	Ni (wt. %)	Fe (wt. %)
0	22.2321	36.18	61.39
6	24.4157	16.54	79.29
10	24.8950	15.43	81.30
12	25.6212	31.51	64.83

3.2 Effect of H_3 BO_3 on the Surface Morphology

The surface morphology of Ni-Fe coatings electrodeposited at different boric acid concentration is shown in Fig. 3. It is seen that all the electrodeposited coatings exhibit a uniform surface structure consisting of nodular particles, compact, crack free and include numerous globular grains. The boric acid plays a significant role on the width and density of nodular particles. However, the coatings exhibit tiny nodular particles ranging in size from 22 to 25 nm, resulting in the formation of a more compact surface structure. In addition, from the SEM results, it can be concluded that the variation of H_3BO_3 does not play a significant role on the surface morphology of electroplated Ni-Fe.

3.3 Effect of H_3BO_3 on the Chemical Composition of Coatings

From the results obtained in Table 3, we note that Ni and Fe contents in the coatings vary with an increase in boric acid concentrations. With an increase in



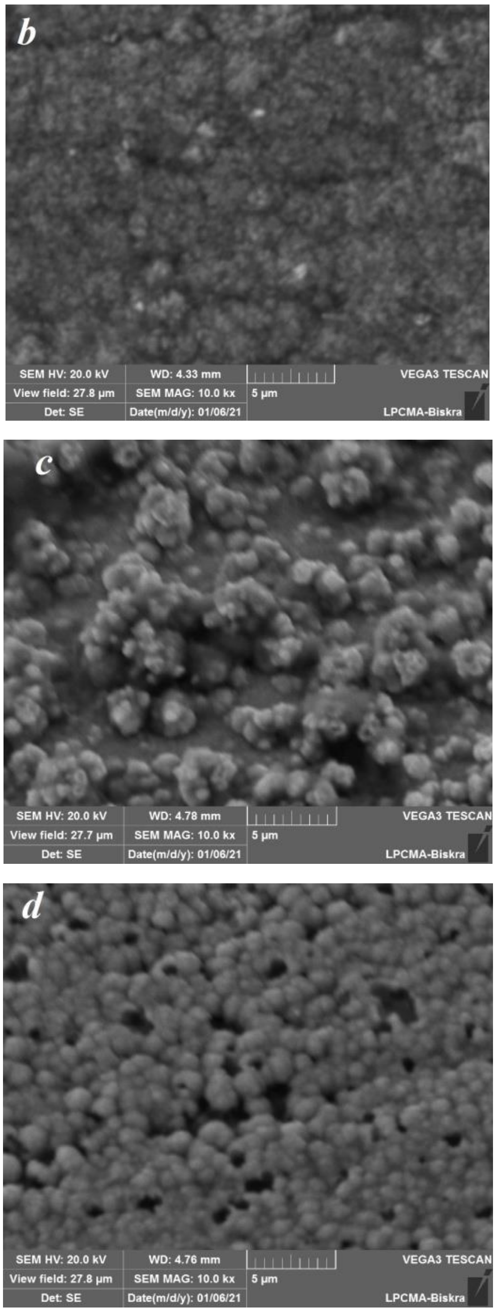


Fig. 3 – Surface morphology of Ni-Fe coatings electrodeposited at different boric acid concentrations: (a) 0 (g/l), (b) 6 (g/l), (c) 10 (g/l), and (d) 12 (g/l)

the boric acid concentration from 0 to 10 g/l, the Fe content in the coatings increases from 61.39 to 81.30 wt. % and then decreases to 64.83 wt. %, and the Ni content in the coatings decreases from 36.18 to 15.43 wt. %, then increases to 31.51 wt. % in 12 g/l of boric acid concentration. The reason for the decrease in the nickel content in the alloy is due to the following. Boric acid acts on the passivation surface of the electrode, which prevents the reduction and deposition of nickel on the cathode, or boric acid acts as a membrane that prevents the passage of nickel. Furthermore, the high content of iron in the alloy is due to its attraction towards the cathode as a result of changing the acid concentration instead of nickel ions, and this is due to the partial restriction of nickel ions by oxygen.

Table 3 – Elemental chemical composition (wt. %) of Ni-Fe coating at different boric acidic concentrations

Conc. (g/l)	Ni (wt. %)	Fe (wt. %)	Cu (wt. %)	O (wt. %)
0	36.18	61.39	0.93	1.50
6	16.54	79.29	0.77	3.40
10	15.43	81.30	0.81	2.45
12	31.51	64.83	2.05	1.62

3.4 Effect of Boric Acid Concentration on the Microhardness of Ni-Fe Coatings

Table 4 shows the relationship between microhardness (*Hv*) and grain size *D* (nm) of Ni-Fe coating. The values are the average of five measurements. This indicates that the hardness decreases as the grain size increases from 128.33 *Hv* to 111.33 *Hv* (inverse proportion). Obviously, for crystalline materials, the hardness increases with decreasing grain size [17]. Moreover, the plastic deformation of the deposit, resulting from the movement of dislocations and sliding of grain boundaries, is the main reason for the deformation.

Table 4 – Effect of boric acid concentration on the grain size and microhardness

Concentration (g/l)	Grain size (nm)	Microhardness (Hv)
0	22.2321	128.33
6	24.4157	127.33
10	24.8950	118.83
12	25.6212	111.33

4. CONCLUSIONS

The experimental results clearly show that boric acid has an effect on the chemical composition, morphological and structural properties of Ni-Fe thin film samples.

1. SEM of electrodeposited Ni-Fe coatings shows that in the absence of boric acid, alloy precipitates are compact and hard. On the other hand, heterogeneous and large-grained alloy precipitates are obtained by adding boric acid.
2. Addition of boric acid to the bath of Ni-Fe coatings improves not only the deposition efficiency but acts as a membrane preventing the passage of nickel.
3. Boric acid can strongly absorb on the cathode to impede proton reduction and may form some complex with iron.
4. The deposit hardness decreases with decreasing nickel content.

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Вплив добавки на властивості покриття Ni-Fe

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Електроосаджені покриття Ni-Fe використовуються в багатьох галузях, таких як корозія, знос, магнітні та електричні застосування та як електрокаталітичні матеріали. Поліпшення властивостей покриттів Ni-Fe та їх широкий спектр можуть бути досягнуті шляхом вибору різних параметрів електроосадження. У роботі покриття Ni-Fe наносяться на мідні підкладки. Для вивчення впливу борної кислоти на морфологію, фазовий склад, вміст нікелю та мікротвердість покриттів використовуються рентгеноструктурний аналіз (XRD), скануючий електронний мікроскоп (SEM), енергодисперсійна спектроскопія (EDS) та аналіз мікротвердості. Рентгенограми отриманих зразків підтверджують наявність гострих піків, які вказують на гарну кристалічність. Морфологія електроосаджених покриттів Ni-Fe показує, що зерна мають сферичну природу для всіх зразків, а аналіз EDS композиту Ni-Fe підтверджує, що відновлення Ni уповільнюється зі збільшенням концентрації борної кислоти. Крім того, зниження концентрації борної кислоти у ванні може призвести до поліпшення властивостей покриття Ni-Fe.

Ключові слова: Покриття Ni-Fe, Морфологія, Борна кислота, XRD.