Research Article

The Physical and Magnetic Properties of Electrodeposited Co-Fe Nanocoating with Different Deposition Times

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Using the electrodeposition process, cobalt-iron (Co-Fe) nanocrystalline coatings were successfully synthesized onto stainless steel in deposition times of 30, 60, and 90 minutes. The temperature used throughout the process was 50°C in an acidic environment of pH 3. By changing the deposition time, physical properties such as phase and crystallographic structure, surface morphology, grain size, microhardness, and magnetic properties of Co-Fe coatings were examined. FESEM micrographs showed that the grain sizes of the coatings were in the range from 57.9 nm to 70.2 nm. Dendrite and irregular shapes were found in the microstructure of Co-Fe nanocoating. The Co-Fe nanocrystalline coating prepared in a deposition time of 90 minutes achieved the highest microhardness of 339 HVN. The magnetic properties associated with Co-Fe nanocoating at longer deposition times show greater coercivity, H_c , and saturation magnetization, M_s , values of 56.43 Oe and 70.45 eμ/g, respectively. The M-H curves for all the Co-Fe coatings exhibited soft ferromagnetic behaviour with narrow hysteresis loops. It was found that increasing the deposition time also improved the microhardness and magnetic properties of Co-Fe nanocoating, which is much needed for long-life high-coercivity magnetic strip card applications.

1. Introduction

Nanoparticles are ultrafine particles with a diameter less than 100 nm which are fundamental in nanocrystalline materials. These materials exhibit enhancing properties, that is, physical, chemical, and mechanical, resulting from a reduction of grain size and existence of interphase within the grain boundaries of the microstructure [1, 2]. Electrodeposition is a viable, low-cost process in synthesizing the nanomaterials. It can be used on metals, alloys, polymers, and composites [3]. It can also produce coatings on various substrates requiring higher deposition rates [4]. Electrodeposition is also suitable for any industrial application since it can produce deposits for restricted areas such as tiny parts in machines or any equipment. In electrodeposition, the properties of the nanocrystalline materials can be improved along with their microstructure being controlled by optimizing the operating parameters such as deposition time, pH, bath temperature, direct current density, and electrolytic composition. A diverse

range of applications has resulted in the interest for nanoparticles with a wide range of magnetic properties. The study of magnetic properties for softer magnetic materials has gained interest among the researchers due to their potential in the manufacturing of components for microelectromechanical system (MEMS) [5]. In magnetic data storage applications, ferromagnetic nanoparticles with a high coercivity, H_c , and saturation magnetization, M_s , is explored for high-density magnetic media such as magnetic recording write heads [6–8]. The magnetic properties such as M_s and H_c of soft magnetic materials are very important to be studied in order to evaluate the performance of those soft magnetic materials.

Previous findings have reported on the magnetic properties, M_s and H_c , of Co-Fe alloys in relation to their different alloy compositions in electrolytes [9]. It was found that the values of M_s and H_c increased linearly with additional contents of Fe. The increase in the M_s value was correlated to the crystal structure or phases of the films. The coexistence of both Co-Fe face-centred cubic (FCC) and Co-Fe

body-centred cubic (BCC) phases in the microstructure was possibly caused by higher M_s values as the Fe content was increased. Additionally, initial research works reported that, for nanocrystalline materials, there was a large reduction in M_s with decreasing grain size [10]. Reference [10] stated a 40% decrease in M_s for nanocrystalline Fe compared to its bulk polycrystalline Fe. It was then concluded that the reduction in M_s was attributed to the differences in the magnetic microstructure between nanocrystalline and conventional polycrystalline Fe.

Other previous paper work reviewed the size effects in nanocrystalline metals and alloys made by electroforming, a particular form of electrodeposition [11]. The main size effects in these nanocrystalline materials came from grain size reduction down to less than 5 nm and down towards the limit of the amorphous structure. Fully dense nanocrystalline metals and alloys have unique microstructures consisting of nanometre-sized crystals and large volume fractions of grain boundary and triple junction defects. It has been shown that several properties such as Young's modulus, thermal expansion, and saturation magnetization were not greatly affected by the presence of high concentrations of these defects. In contrast, properties that depend on the interactions of intercrystalline defects with other structural characteristics such as dislocations, precipitates, and electrons show significant grain size effects. These include hardness, wear resistance, electrical resistivity, and coercivity. In addition, the introduction of structural disorder by grain boundaries and triple junction had an insignificant effect on M_s compared with the chemical disorder introduced by additional alloying [12].

The study on the characterization and magnetic properties of electrodeposited Co-Fe alloys with a citrate stabilized sulphate bath was reported elsewhere in relation to the plating conditions and structure parameters [13]. The influence of ammonium citrate dosage, Fe content, and plating temperature was investigated in relation to magnetic properties. A lower value of H_c (10 Oe) was obtained with a low dosage of citrate bath (2–20 g/L) due to the low stress in Co-Fe film [14]. The value of H_c of the deposited film decreased as the Fe content reached the minimum requirement of approximately 40 atomic% Fe. However, the H_c value slowly rose as the Fe content increased from the minimum content. Similarly, the H_c value became greater with increments in the plating temperature. An increase in deposition temperature was most likely due to an increase in deposit grain size, which should have increased the H_c value. The H_c is directly proportional to the deposited grain size [15]. The composition of Co-Fe film with high Fe content and less Co content produced the best soft magnetic properties— H_c value of 17 Oe, which is still acceptable, and an M_s value of 2.04 T, which is quite good. The addition of ammonium citrate in the deposition bath was believed to have not sacrificed the magnetic properties of the Co-Fe films. The lowest H_c value of 10 Oe, which was obtained in a low dosage of ammonium citrate, corresponded to a mixture of FCC and BCC phases with BCC phase denominating. The optimal soft magnetic properties of lower H_c and higher M_s were produced when both phases, BCC and FCC were codeposited in the Co-Fe microstructure. This is

TABLE 1: Molar concentration of precursors in the electrolyte composition for the synthesis of Co-Fe nanocoating.

Precursor	Molar concentration, molarity (M)
CoSO ₄	0.075
FeSO ₄	0.03
H ₃ BO ₃	0.4
Sodium saccharine	0.01

due to the crystallization competition between the BCC and FCC phases which led to the reduction in grain size [15].

Thus, in this research work, the synthesis of Co-Fe nanocoatings using electrodeposition method on stainless steel substrate was reported. The physical and magnetic properties of as-synthesized Co-Fe deposits were investigated in relation to the different deposition times of 30, 60, and 90 minutes.

2. Experimental Work

The electrolyte used in the electrodeposition process was a mixture of CoSO₄, FeSO₄, and H₃BO₃ with an organic additive of saccharine [16]. Table 1 represents the molar concentration of the precursors in the bath composition. The Co-Fe coatings were deposited on the stainless steel substrate while a graphite electrode was used as the anode. A Co-Fe nanocrystalline was deposited with a flow of direct current of density 0.08 A/cm². The pH of the electrolyte was maintained at 3.0. The Co-Fe coatings were formed in 30-, 60- and 90-minute deposition times. All the operating parameters such as temperature, pH, direct current density, and deposition time during the electrodeposition process were controlled and maintained. Each experiment was carried out with a freshly prepared solution. The equipment used to test the characteristics of as-synthesized Co-Fe coatings were ULTIMA IV FD 3668N, X-ray diffractometer (XRD), and JEOL JSM-7600F, a Field Emission Scanning Electron Microscope (FESEM). Hardness of the coating sample was measured with a MITUTOYO MVK-H1, Vickers Microhardness Tester using a load of 200 gm. The Alternating Gradient Magnetometer, AGM (Model 2900) was used to determine the magnetic properties of Co-Fe coatings. Figure 1 illustrates the schematic diagram of the electrodeposition process for the synthesis of Co-Fe nanocoating.

3. Results and Discussion

3.1. Phase and Crystallographic Structure Characterization. XRD measurements of as-synthesized Co-Fe coatings were carried out from 30° to 100° 2θ angle. Figure 2 shows the XRD patterns of the electrodeposited Co-Fe coatings at different deposition times of 30, 60, and 90 minutes. The XRD spectrum for the Co-Fe phase reveals the characteristic peaks at 2θ angles of 45°, 65°, and 83°. The peaks correspond to (110), (200), and (211) planes. The Co-Fe phase is identified as the BCC crystal structure with JCPDS no. 10717173. Observing the XRD spectrum for pure cobalt during depositions of 30 and 60 minutes, the Co phase was observed at 2θ angles

FIGURE 1: Schematic diagram of electrodeposition process.

FIGURE 2: X-ray diffraction patterns for three Co-Fe nanocoatings obtained in 30-, 60-, and 90-minute depositions.

of 75° and 91° with (110) and (200) planes, respectively. Pure Co phases have the hexagonal close packed (HCP) crystal structure similar with JCPDS no. 50727.

Sulphur (S) peaks were observed in all Co-Fe XRD patterns prepared in three deposition times. The S peaks, which are indexed at 2θ angles of approximately 44° and 51° , have the respective corresponding planes of (2210) and (266). S phases have similar orthorhombic crystal structure as with JCPDS no. 80247. As for 90-minute depositions; the S phase reveals a characteristic peak at 2θ angle of 51° which disappeared and left only one S phase at 44° .

Saccharine when used as an organic additive in the electrodeposition process of Co-Fe nanocoating was the main source for the S element in the microstructure. The incorporation of the S element in the Co-Fe microstructure is believed to have occurred due to the two independent mechanisms. There are saccharine electroreduction and physical incorporation of saccharine molecules during the deposit growth. This incorporation of S elements in the Co-Fe microstructure was also reported in [17]. Referring to Figure 2, the peak of pure Co is fully dissolved in a 90-minute deposition which showed that the Co-Fe phase was completely formed.

3.2. Surface Morphology Characterization. Figure 3 shows the granular structure of Co-Fe coatings in three different deposition times: 30, 60, and 90 minutes. All Co-Fe coatings have dendritic and irregular microstructures. The grains resemble the pine tree-like shape which is known as dendrite, meaning branching. Secondary dendrite arms branch off the primary arm and tertiary arms off the secondary arms, and so forth. The formation of dendrite occurs because the grains are developed in defined planes due to the created crystal lattice [18]. The dendritic microstructure which contains Co-rich content is mostly found in the Co-Fe coating that was deposited for 30 minutes. This was due to the existence of pure Co phases compared to the other deposition times. This is in agreement with the XRD results. The oriented-attachment mechanism is assumed to have occurred in Co-Fe nanoparticle microstructures which caused the formation of dendritic microstructures [19]. In contrast, the majority of the microstructure area in the 90-minute deposition was overwhelmed with the existence of a greater number of irregular grains. These irregular grains are merely grains without a smooth surface because its growth was impeded by contact with another grain or boundary surface. The interface formed between grains is called the grain boundary. The atoms between the grains at the grain boundaries have no crystalline structure and are arranged in a disordered fashion [18]. In conclusion, the presence of irregular grains in the microstructure develops an interface between grain boundaries. Whereas, the grains with dendritic morphology have developed pine tree-like shapes in the microstructure, each with branches that have defined crystal planes.

The average grain sizes decreased from 70.2 nm to 57.9 nm with deposition times increasing. It is believed that there is insufficient time for the grains to distribute and therefore leave certain substrate areas empty in the microstructure of Co-Fe nanocoating for 30-minute depositions. The phenomenon has led to the creation of underdeveloped dendritic structures and bigger grain size due to the distribution of the Co-Fe atoms being mostly inside the grains compared to the grain boundaries. Agglomeration and grain growth tend to happen in 60-minute depositions as shown in the Figure 3(b). The atoms in the microstructure are fused, combining with each other and producing larger grains. Meanwhile, the irregular grains are smaller in FESEM micrographs of 90-minute electrodepositions compared to 30- and 60-minute depositions as shown in Figure 3(c).

In the 90-minute deposition, the Co-Fe atoms have ample time for rearranging themselves in the microstructure as compared to other's deposition times. For the rearrangement of atoms occurring in 90-minute depositions, a large amount of Co-Fe atoms have the opportunity to be located around the grain boundaries with a more complicated and disorderly manner. There are restrictions in their arrangement imposed by the adjacent crystal lattice of disoriented grains in the microstructure [20]. These Co-Fe atoms are arranged with a variety of interatomic spacing differing from boundary to boundary [2]. Furthermore, a greater number of grain boundaries with the highest proportion of atoms inside the boundaries have created an extremely high fractional volume

FIGURE 3: FESEM micrographs of Co-Fe nanocoatings deposited in (a) 30, (b) 60, and (c) 90 minutes.

of grain boundary phases in the Co-Fe microstructure. Therefore, a greater volume fraction of grain boundary phases has developed the fully compact and dense Co-Fe microstructure which has resulted in a reduction in grain size and enhanced properties from the bulk materials. In addition, the Co-Fe microstructure is also believed to be fully completed in this deposition time since there is no presence of pure Co phases in the microstructure as can be seen in the XRD result. As a conclusion, higher proportion of boundary atoms in grain boundaries compared to those inside the grains has created the fully dense and compact microstructure. These high volume fractions of grain boundaries lead to the reduction of grain size.

3.3. Microhardness Measurement. The microhardness measurements were taken from an average of 10 measurements using a 200 gm load. The microhardness values of as-synthesized Co-Fe coated samples in 30-, 60-, and 90-minute depositions were 287, 291, and 339 HVN, respectively, as shown in Figure 4. The highest microhardness was found at a deposition time of 90 minutes. This was with the smallest grain size (57.9 nm) and had an existence of structure compactness and grain boundaries in the coating microstructure. This is in parallel with the FESEM result. Moreover, the existence of these grain boundaries prevents the dislocation motion by changing the direction or stopping the movement of the dislocation. Consequently, the change in the dislocated direction movement results in a harder material. This result

FIGURE 4: The average microhardness of Co-Fe coatings at different deposition times of 30, 60, and 90 minutes.

is consistent with [21]. It can be concluded that as the deposition time increases, the average microhardness of the Co-Fe coatings increases.

3.4. Magnetic Properties. Figure 5 shows the magnetic properties of as-synthesized Co-Fe coatings at different deposition times of 30, 60, and 90 minutes in M-H hysteresis loops. The M-H curve of Co-Fe coatings passes the origin of the graph. All the M-H curves illustrate that the electrodeposited Co-Fe coatings exhibit the soft ferromagnetic behaviour due to the presence of narrow and smaller well-defined hysteresis loop areas which can be seen in Figure 4. This similar finding

FIGURE 5: M-H hysteresis loops of Co-Fe nanocoatings prepared in deposition times of (a) 30, (b) 60, and (c) 90 minutes.

TABLE 2: The magnetic properties of electrodeposited Co-Fe coatings in three different deposition times.

Deposition time (minutes)	M_s (e μ /g)	H_c (Oe)
30	22.99	42.56
60	47.87	52.67
90	70.45	56.43

was reported in [22]. The Co-Fe nanoparticles in 90-minute depositions show a broader hysteresis loop area compared to the other samples indicated by the higher M_s value.

The magnetic properties of electrodeposited Co-Fe coatings for different deposition times are tabulated in Table 2. The M_s of Co-Fe coating increases with longer deposition times from 22.99 to 70.45 e μ /g. This increase in M_s value can be explained by the change in grain shape from dendritic to irregular for longer deposition times [5]. The highest proportion of the irregular grains in the 90-minute deposition of the Co-Fe nanocoating developed a compact and fully dense microstructure with smaller grain sizes. The reduced grain size and structure compactness have resulted from a high volume of disorderly arranged atoms in the grain boundaries as compared to the grains themselves. This phenomenon has led to the increase of M_s [23]. The formation of a compact dendritic morphology in the crystallized Co-Fe-based alloy was believed to have given an undesirable effect towards the soft magnetic properties [24].

The value of H_c was found to be greater with the grain size reducing as deposition times increased. The increase of H_c is usually influenced by larger grain sizes [23] but was not consistent with the results of this research. Additionally, based on research work taken from [25, 26], low H_c was found in the Co-Fe FCC + Co-Fe BCC region. However, in 30-minute depositions, low H_c value cannot be related to the creation of combined FCC + BCC phases since Co-Fe

phases exhibit pure BCC crystal structures. Therefore, further research should be conducted to investigate this phenomenon of H_c increment.

4. Conclusion

The Co-Fe phase in the Co-Fe coatings has a BCC crystal structure. Morphology studies reveal that the granular structure of Co-Fe coatings consist of a dendritic and irregular microstructure. The average grain size for all Co-Fe coatings is between 57.9 nm and 70.2 nm with the smallest grain size being noted in a deposition time of 90 minutes. The smallest grains produced the highest microhardness of 339 HVN. The mechanical properties of microhardness are not affected by the presence of phases in the Co-Fe microstructure. On the other hand, this mechanical property is closely correlated to the reduction in grain size and interactions of intercrystalline defects, that is, grain boundaries with other structural characteristics which include dislocations in the Co-Fe microstructure.

All the Co-Fe coatings show soft ferromagnetic behaviour with narrow hysteresis loops. The M_s and H_c are increased as the deposition time increases. M_s of electrodeposited ferromagnetic materials such as Co-Fe nanocoating are generally independent of electroplating conditions. It is only dependent on film compositions (Fe and Co content), phases and crystallographic structures (BCC phase), and surface morphologies (irregular and dendritic structures) in the microstructure. Usually, H_c values are directly proportional to the grain size and are affected by the presence of intercrystalline defects of grain boundaries and triple junctions. The best optimal magnetic properties which have reduced H_c and higher M_s are related to the coexistence of Co-Fe BCC and FCC phases in the microstructure. However, in this research, with the existence of only pure Co-Fe BCC phases, higher M_s and H_c values were found during depositions times of 90 minutes.

Effects on grain size reduction are directly proportional to the increase of M_s and H_c . It is suggested that the nanocrystalline Co-Fe coatings prepared at 90 minutes produce better H_c and hardness due to smaller grain size.

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