

Research Article

Comparison of Microstructural and Morphological Properties of Electrodeposited Fe-Cu Thin Films with Low and High Fe : Cu Ratio

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Fe-Cu films with low and high Fe : Cu ratio have been produced from the electrolytes with different Fe ion concentrations at a constant deposition potential of -1400 mV versus saturated calomel electrode (SCE) by electrodeposition technique onto indium tin oxide (ITO) coated conducting glass substrates. It was observed that the variation of Fe ion concentration in the electrolyte had a very strong influence on the compositional, surface morphological, and microstructural properties of the Fe-Cu films. An increase in the Fe ion concentration within the plating bath increased the Fe content, consequently Fe : Cu ratio within the films. The crystallographic structure analysis showed that the Fe-Cu films had a mixture of face-centered cubic (fcc) Cu and body centered cubic (bcc) α -Fe phases. The average crystallite size decreased with the Fe ion concentration. The film electrodeposited from the electrolyte with low Fe ion concentration exhibited a morphology consisting of dendritic structures. However, the film morphology changed from dendritic structure to cauliflower-like structure at high Fe ion concentration. The surface roughness and grain size were found to decrease significantly with increasing Fe ion concentration in the electrolyte. The significant differences observed in the microstructural and morphological properties caused by the change of Fe ion concentration in the electrolyte were ascribed to the change of Fe : Cu ratio within the films.

1. Introduction

The fabrication of electrodeposited thin film multilayers or granular alloys consisting of magnetic and nonmagnetic metals has attracted great interest due to their technological applications [1]. Among them, Fe-Cu films find applications in electronic industry [2]. Electrodeposition is a versatile growth process for the fabrication of metallic thin film materials, which find applications in electronics, magneto-electronics, magnetic recording, and microsystems industries due to its some unique features [3-7]. Aside from this, electrodeposition process also provides the deposition under ambient temperature and pressure and does not require relatively expensive equipment [6, 8]. It is well known that a small change in the growth parameters has a strong influence on the properties of the electrodeposited films.

It has been shown that the composition of electrolyte is one of the most important growth parameters affecting strongly the properties of the electrodeposited thin film materials deposited onto indium tin oxide (ITO) coated glass substrates [9-11]. In previous works, we investigated the properties of electrodeposited Fe-Cu films grown onto ITO coated glass substrates at different electrolyte temperatures and current densities [12, 13]. However, the effect of Fe : Cu ratio caused by the change of Fe ion concentration in the electrolyte on the structural and morphological properties of Fe-Cu films prepared onto ITO coated glass substrates has not been reported. The surface morphology of the films produced by electrodeposition technique is critically important and it can be controlled by varying the deposition parameters. Therefore, in this research, electrodeposited Fe-Cu films with low and high Fe : Cu ratio were produced from

the electrolytes with low and high Fe ion concentrations onto ITO coated glass substrates under potentiostatic conditions. The variations of the microstructures, compositions, and morphologies of the films were analyzed with respect to Fe ion concentration in the electrolyte. In this study, it is shown that the Fe ion concentration in the electrolyte has a significant effect on the microstructural, compositional, and surface morphological properties of electrodeposited Fe–Cu thin films.

2. Materials and Methods

Fe–Cu specimens were grown from the electrolytes composed of low, intermediate, and high Fe ion concentrations (0.02, 0.04, and 0.08 M, resp.), but the same Cu sulfate (0.01 M) and boric acid (0.1 M) concentrations. All chemicals were dissolved in deionized water. The bath temperature and pH were $25 \pm 1^\circ\text{C}$ and 3.8 ± 0.1 , respectively. Although the pH of the electrolytes decreased during the electrodeposition process of the films, we did not use any additive in order to obtain the stable electrolyte pH because of its possible effect on the electrodeposition process. For the deposition process, the electrodeposition system consisting of a potentiostat/galvanostat (VersaSTAT 3) with a conventional three-electrode electrochemical cell was used. Throughout the electroplating process, a saturated calomel electrode (SCE) was served as the reference electrode. A platinum wire and indium tin oxide (ITO) coated conducting glass substrate were used as counter electrode and working electrodes, respectively. The working area was about 0.8 cm^2 . The ITO electrodes were cleaned with acetone followed by ethanol and then ultrasonically rinsed for 10 min in water before deposition of the films. In all cases, the films were produced at a constant deposition potential of -1400 mV versus SCE, and the electroplating time was controlled to obtain equal cathodic charge. In the present study, the thickness of all films was set around 500 nm.

The structure of Fe–Cu deposit samples was characterized by means of Panalytical–Empyrean XRD diffractometer using $\text{CuK}\alpha$ radiation ($\lambda = 0.154059\text{ nm}$). The XRD data were recorded in a range from 42° to 54° with a step width of 0.02° . The elemental constitutions were analyzed using an energy dispersive X-ray spectroscopy (EDX). The surface morphological structure of resultant films was studied with the help of Zeiss Supra 50 Vp model SEM and AFM (Veeco Multimode V) techniques. To avoid possible oxidation of the surfaces, surface topographic images were obtained immediately after the production process at ambient temperature and pressure.

3. Results and Discussion

The electrodeposition of Fe–Cu films has been carried out from the electrolytes containing low, intermediate, and high Fe ion concentrations. Firstly, the potentiostatic current–time transients for the Fe–Cu films electrodeposited from electrolytes with low and high Fe ion concentrations were employed as a function of electroplating time during the

electroplating process in order to study the stability of electrodeposition of the Fe–Cu films. The potentiostatic current–time transients of the films grown from the electrolytes with low and high Fe ion concentrations recorded for the first 50 s of electroplating process are illustrated in Figure 1. It is clearly seen that the current density remains almost stable, indicating that the proper films are electrodeposited from the electrolytes with different Fe ion concentrations at a constant deposition potential. As can be seen from Figure 1, the current density was also observed to decrease towards more negative values as the Fe ion concentration in the electrolyte increased.

As a second step of this investigation, the elemental analysis of the films was done using EDX with respect to Fe ion concentration in the electrolyte. The film produced from the electrolyte with low Fe ion concentration consists of 33.5 at.% Fe and 66.5 at.% Cu. However, for the film electrodeposited from the electrolyte with high Fe ion concentration, the composition is 80.2 at.% Fe and 19.8 at.% Cu. These results reveal that an increase in the Fe ion concentration in the electrolyte increases the Fe:Cu ratio within the films. As mentioned above, the value of the current density decreased towards more negative values with increasing Fe ion concentration in the electrolyte. Thus, a decrease in the current density towards more negative values with the Fe ion concentration in the electrolyte leads to Fe–Cu film with higher Fe:Cu ratio. It has been shown that the Fe–Cu films are Fe-rich as the films are produced at more negative current densities [13]. On the other hand, the ratio of Fe:Cu within the films is found to be lower than the ion concentration ratio of $\text{Fe}^{+2}:\text{Cu}^{+2}$ in the electrolyte. The possible reason for this effect may be preferentially Cu reduction process within the electrolyte containing the Fe and Cu ions, since Cu is a more noble metal [14]. Consequently, according to results obtained from the EDX measurements, it can be obviously said that the Fe:Cu ratio within the films is affected significantly by the Fe ion concentration in the electrolyte.

XRD measurements were performed in order to study the effect of Fe ion concentration on the crystal structure of the electrodeposited Fe–Cu films. XRD patterns of Fe–Cu films electrodeposited from the electrolytes with low and high Fe ion concentrations are presented in Figure 2. As the fcc Cu peak positions are distinct from those of bcc Fe, it is easy to determine and compare the bcc versus fcc phase in the film [15]. In the XRD patterns, diffraction peaks related to the fcc Cu (111), fcc Cu (200), and bcc Fe (110) are observed irrespective of Fe ion concentration studied in the experiment. These results obtained from the XRD patterns pointed to the coexistence of fcc Cu (111) and bcc α -Fe (110) phases. It has been shown that the structure of the Fe particles in Cu matrix is strongly related to the Fe content depending on the growth technique [16, 17]. In equilibrium, the Fe and Cu elements have very small mutual solid solubility, and the equilibrium Fe–Cu phase diagram does not exhibit intermediate phases [18]. The iron is incorporated in the fcc–Cu structure, giving rise to a Cu-based fcc Fe–Cu solid solution for high Cu content. A single phase bcc structure is obtained for Fe–Cu films for high Fe content. However, the films show mixed fcc and bcc phase in intermediate

composition range [15–17]. In electrodeposited Fe–Cu films, the structure of Fe grains in Cu matrix is bcc as the Fe content within the films is higher than 30%, while the structure of them is fcc for the Fe content lower than 27% [19]. Thus, in the present study, it is concluded that the Fe–Cu films electrodeposited onto ITO coated glass substrates from the electrolytes with low and high Fe ion concentrations consist of a mixture of fcc Cu and bcc Fe particles with respect to their Fe content. These results obtained about our electrodeposited Fe–Cu films are in good agreement with the results found in previous published papers [12, 13, 20]. On the other hand, it is found from the XRD patterns given in Figure 2 that the relative intensity of bcc Fe (110) diffraction peak increases with the Fe ion concentration in the electrolyte. In opposition to that, a decrease in the relative intensity of fcc Cu (111) and Cu (200) diffraction peaks with increasing Fe ion concentration in the electrolyte is also observed, as can be clearly seen from Figure 2. These differences observed in the structural properties can be ascribed to the increase of Fe : Cu ratio within the films with increasing Fe ion concentration in the electrolyte.

The average crystallite size in the direction perpendicular to the plane of the sample was analyzed by application of Scherrer's formula [21]. The increase of Fe ion concentration in the electrolyte induces change in the full width at half maximum (FWHM) of the diffraction peaks. The average crystallite sizes for the bcc Fe (110) peaks are determined to be 35 and 26 nm for the films electrodeposited from the electrolytes with low and high Fe ion concentrations, respectively, indicating a nanocrystalline structure of the films. This result also reveals that the average crystallite size of the films decreases with increasing Fe ion concentration in the electrolyte. As seen in Figure 1, the current density decreased towards more negative values as the Fe ion concentration within the electrolyte increased. Thus, it is expected that a decrement of the current density towards more negative values observed at high Fe ion concentration increases the nucleation densities and consequently reduces the crystallite size [13, 22–25].

SEM measurements were performed to characterize the effect of Fe ion concentration on the surface morphology of Fe–Cu films. It is observed that the electrodeposited Fe–Cu films prepared from the electrolytes with low and high Fe ion concentrations have also different morphological features. SEM topographic images of electrodeposited Fe–Cu films grown from the electrolytes with low and high Fe ion concentrations are shown in Figure 3(a) and b, respectively. Figure 3(c) shows the SEM image of Fe–Cu film electrodeposited from the electrolyte with high Fe ion concentration at lower magnification. It is clearly seen that the surfaces of the films exhibit a compact and crack-free morphology. As shown from Figure 3, there is notable change in the surface morphology of Fe–Cu films with respect to Fe ion concentration in the electrolyte. It is also clearly seen that the film grown from the electrolyte with low Fe ion concentration exhibits a morphology having dendritic structures (Figure 3(a)). The dendritic growth observed in the electrodeposited Fe–Cu film prepared from the electrolyte with low Fe ion concentration may be attributed to

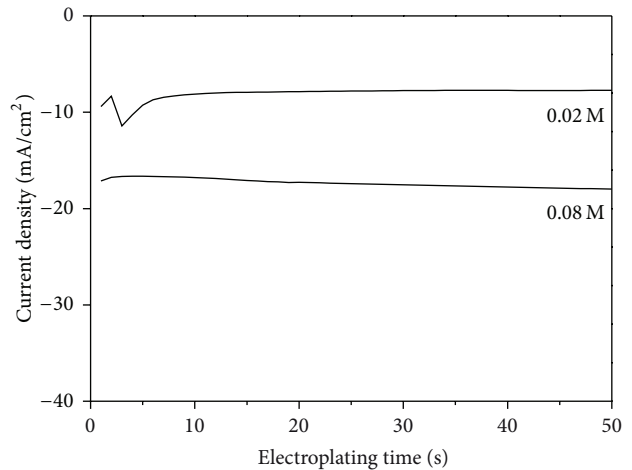


FIGURE 1: Potentiostatic current–time transients of the Fe–Cu films electrodeposited from the electrolytes with low and high Fe ion concentrations.

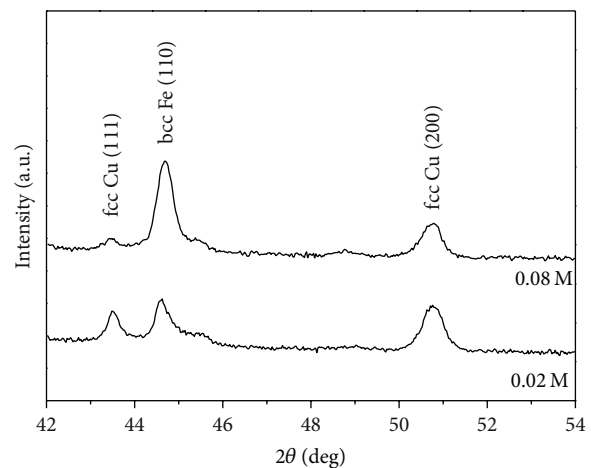


FIGURE 2: XRD patterns of electrodeposited Fe–Cu films prepared from the electrolytes with low and high Fe ion concentrations.

the high Cu content within the film. Dendritic growth of electrodeposited films with high Cu content was ascribed to the diffusive growth mechanism of Cu [14, 26]. On the other hand, on the surface of the film grown from the electrolyte with high Fe ion concentration, the dendritic structures disappear (Figure 3(b)). Such a disappearance of the dendritic structures for the film deposited from the electrolyte with high Fe ion concentration may be due to the change of major component of the film from Cu to Fe as confirmed by the EDX measurements. It can be clearly seen that the surface of the film prepared from the electrolyte with high Fe ion concentration is smoother and more uniform than that of the film grown from the electrolyte with low Fe ion concentration (Figure 3(b)); however, the cauliflower-like structures formed due to the agglomeration of the smaller grains are also observed in some regions on the surface as seen from Figure 3(c). According to results obtained from the SEM and EDX analyses, it is concluded that the surface

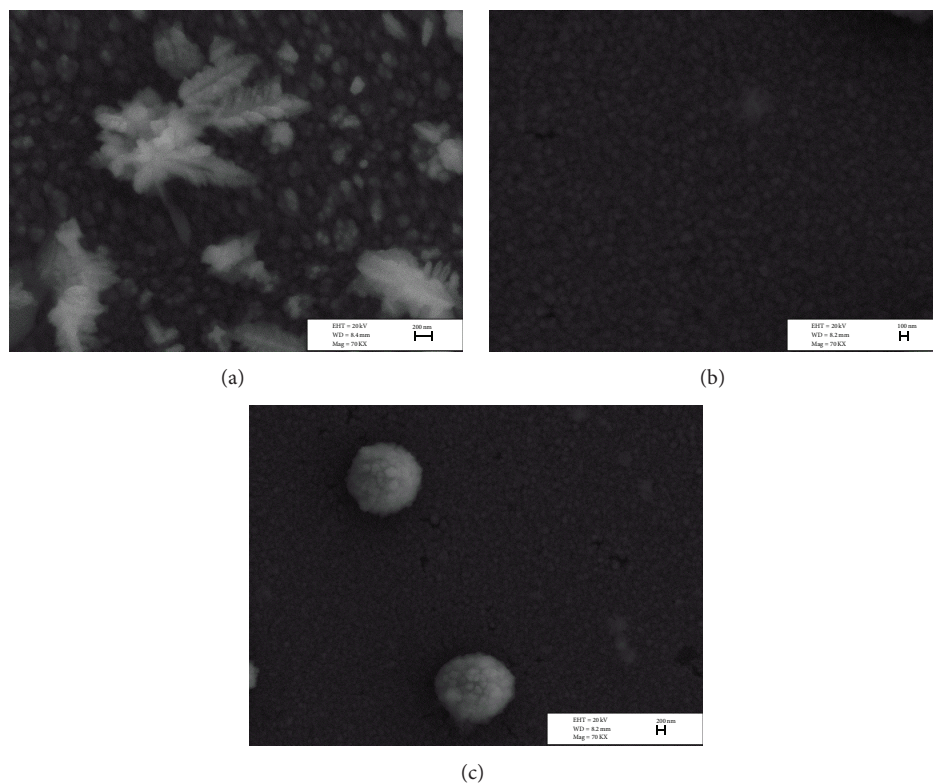


FIGURE 3: SEM topographic images of Fe–Cu films electrodeposited from the electrolytes with low and high Fe ion concentrations (a) 0.02 M, (b) 0.08 M, and (c) 0.08 M at lower magnification, respectively.

morphological structure of electrodeposited Fe–Cu films is affected strongly by the change of Fe:Cu ratio within the films caused by the change of Fe ion concentration in the electrolyte. The film morphology changes from a dendritic structure to a cauliflower-like structure by the increase of Fe:Cu ratio within the films. Similar changes in the surface morphology have been also observed in very recent studies performed about electrodeposited Fe–Cu films prepared at different growth conditions and have been also attributed to the compositional differences [12, 13].

Significant changes observed in the surface morphology with the Fe:Cu ratio may be also related to the amount of hydrogen evolution reaction. As shown in Figure 1, the current density is more negative as the Fe–Cu film is produced from the electrolyte with higher Fe ion concentration. At more negative current densities and/or more negative cathode potentials, the hydrogen evolution reaction takes place and it is vigorous enough to change the electrochemical conditions at the vicinity of the cathode, which gives rise to change of morphological structure [27, 28]. Thus, it is concluded that the reason of the differences observed in the morphological structure with respect to Fe:Cu ratio may be also attributed to the increase of the hydrogen evolution reaction with increasing Fe ion concentration in the electrolyte. In recent studies, the changes of surface morphology of metal films produced by electrodeposition technique at different growth conditions have been also related to the amount of hydrogen evolution reaction [29–33].

The surface morphology of electrodeposited Fe–Cu films prepared from the electrolytes with different Fe ion concentrations was also studied using an AFM. Two-and-three dimensional AFM images over a scan area of $1\ \mu\text{m} \times 1\ \mu\text{m}$ and one-dimensional line scans of the films deposited from the electrolytes with low, intermediate, and high Fe ion concentrations are shown in Figures 4(a), 4(b), and 4(c), respectively. Fe–Cu films exhibited granular growth morphology irrespective of Fe ion concentration as seen in Figure 4. The one-dimensional line scans given also in Figure 4 show that the height and the width of the grains are considerably affected by the Fe ion concentration in the electrolyte. It is clearly seen that both the height and the width of the grains decrease as the Fe ion concentration in the electrolyte increases. Accordingly, it can be concluded that an increase in Fe ion concentration in the electrolyte leads to higher nucleation density and thus encourages the formation of smaller grains on the film surface. The AFM image shown in Figure 4(c) for the film grown from the electrolyte with high Fe ion concentration confirms the existence of the smaller grains. It is well known that the surface roughness of electrodeposited thin films is generally related to the grain size [34]. The variation of the average and the root-mean square (RMS) surface roughness in Fe–Cu films as a function of Fe ion concentration in the electrolyte is illustrated in Figure 5. The root-mean square (RMS) surface roughness values are determined to be 71.4, 43, and 18.2 for the films electrodeposited from the electrolytes with low, intermediate, and high Fe ion concentrations,

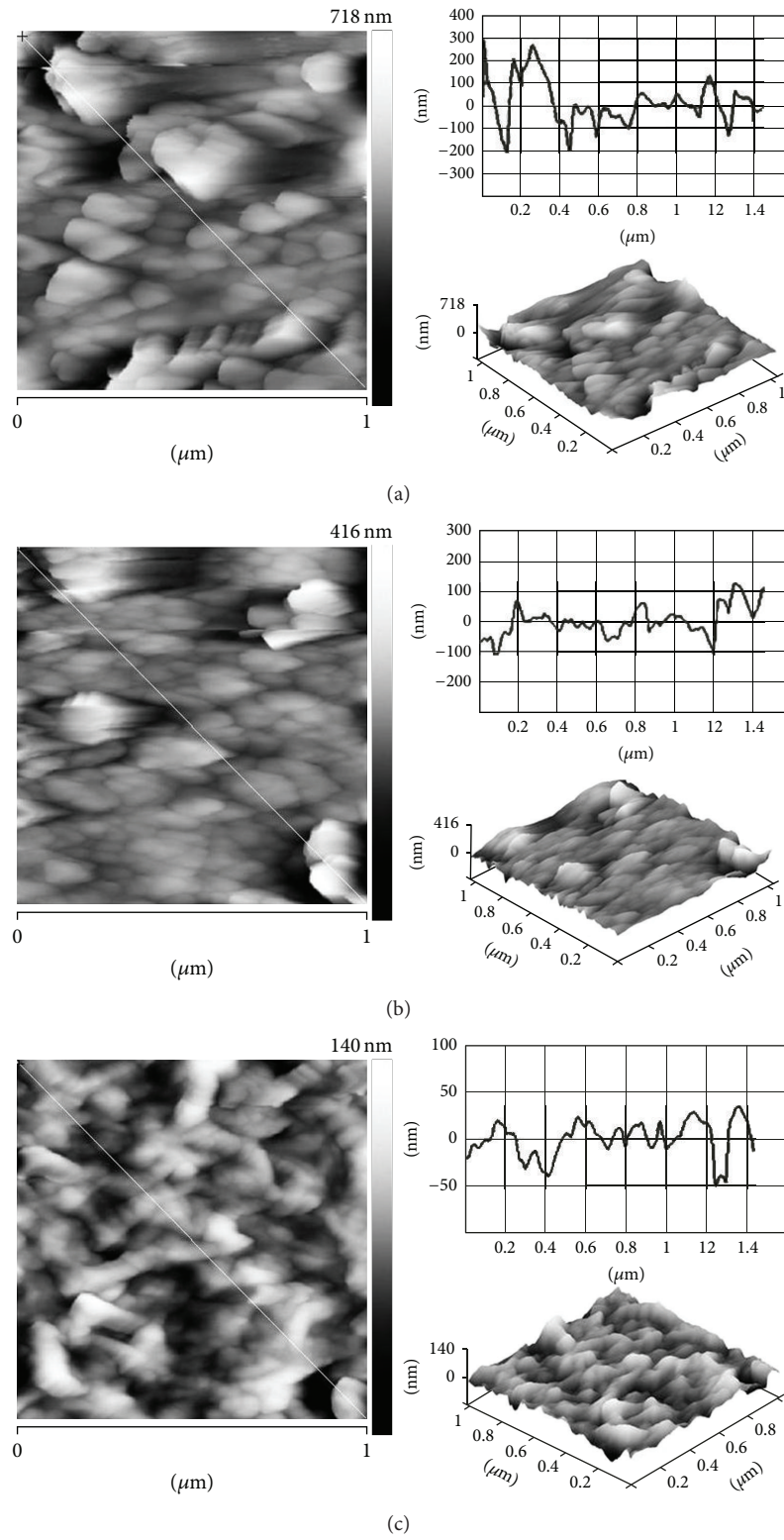


FIGURE 4: Two- and three-dimensional AFM topographic images and one-dimensional line scans of electrodeposited Fe-Cu films grown from the electrolytes with low, intermediate, and high Fe ion concentrations (a) 0.02 M (b) 0.04 M, and (c) 0.08 M, respectively.

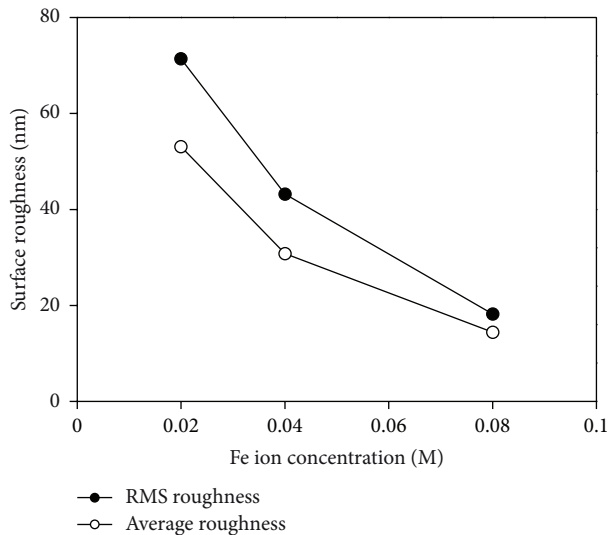


FIGURE 5: The variation of the average and the rms surface roughness in Fe-Cu films as a function of Fe ion concentration in the electrolyte. All lines are guide for the eye.

respectively, indicating the surface roughness of the films decreases strongly with increasing Fe ion concentration in the electrolyte. As a result, the surface roughness of the films decreases significantly with the increase of Fe ion concentration in the electrolyte, and finally the film exhibits a morphology having of smaller grains.

4. Conclusions

We examined and reported-the-variations of the microstructures, compositions, and morphologies of electrodeposited Fe-Cu films with low and high Fe:Cu ratio synthesized onto ITO coated glass substrates from the electrolytes with different Fe ion concentrations. The variation of the Fe ion concentration in the electrolyte changed the Fe:Cu ratio within the films and, hence strongly affected the microstructural and morphological properties of the films. XRD results showed the existence of separated fcc Cu and bcc Fe phases. The relative intensity of the fcc Cu (111) and Cu (200) peaks decreased, whereas the relative intensity of bcc Fe (110) peak increased as the Fe ion concentration in the electrolyte increased. The average crystallite size of the films decreased as the Fe ion concentration in the electrolyte increased. At low Fe ion concentration, the dendritic structures on the film surface were observed; however, the cauliflower-like structures appeared on the surface of the film at high Fe ion concentration due to the change of the Fe : Cu ratio within the films. Fe-Cu film electrodeposited from the electrolyte with high Fe ion concentration exhibited a relatively smooth surface. At low Fe ion concentration, the surface become rougher. The RMS surface roughness was found to decrease significantly from 71.4 to 18.2 nm with increasing Fe ion concentration in the electrolyte. There was also a significant decrease in average grain size as the Fe ion concentration in the electrolyte increased, and finally the film surface

morphology consisted of smaller grains. Accordingly, the significant differences observed in the morphological and microstructural properties might be related to the change of Fe : Cu ratio within the films, which can be controlled by the change of Fe ion concentration in the electrolyte.

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