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The Controllable Growth of CoNiCu Nanoalloy Electrodeposited from Electrolyte-Containing Alkyl Polyglucoside

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Abstract

A controlled growth characteristic of CoNiCu nanoalloys on indium tin oxide coated glass (ITO) has been prepared by alkyl polyglucoside (APG) assisted electrodeposition. The FESEM analysis carried out on the as-prepared samples found that the morphology of nanoscale Co-Ni-Cu alloy particles was strongly influenced by the APG surfactant in the reaction. In a typical process, the morphology of Co-Ni-Cu particles was spherical with excellent size distribution of average size ca. 50 nm. It is in total contrast with those prepared in the absence of APG, of which is irregular shape particles of size ca. 100 nm as the dominant product. The composition analysis on the deposits found that the addition of the APG surfactant in the reaction may modify the ratio of the alloys to some extents, as the change in the co-electrodeposition potential become improbable in the system without the APG surfactant.

Keywords: *Nanoalloys, CoNiCu, Electrodeposition, Alkyl polyglucoside.*

1. INRODUCTION

CoNiCu alloys have attracted active attention in materials chemistry synthesis due to their potential applications for hydrogen evolution reaction electrocatalyst [1], non-enzymatic glucose sensor [2] giant magneto-resistive read heads [3], and magnetic data storage [4–7]. A relatively high corrosion resistance of the CoNiCu [8,9] is known as an advantage which makes these alloys usable over a long period of time. For such applications, CoNiCu properties can be improved by adjusting their chemical composition and preparing the alloys as nano-sized particle [10–12]. In the last two decades, CoNiCu alloys were synthesized using mechanical alloying [10], microemulsion [13]**,** spray pyrolysis [14] and powder metallurgy [15]**.** These methods were successfully employed to control chemicals composition and particles size of Co-Ni-Cu. However, since most applications required the alloys to be prepared a thin layer material on a solid substrate, a technique that can grow the Co-Ni-Cu directly on a substrate is highly demanded. Electrodeposition is one of a prospective method for growing Co-Ni-Cu film directly on the surface [16]. This technique was recognized as a versatile bottom-up method that can produce films with controlled morphology, density as well as chemical composition of which can be achieved by a simple adjustment in the electrodeposition parameters, such as current density and potential [17–20].

However, there is a several major drawback in the electrodeposition of nano-particles onto the substrate surface, such as uncontrolled morphology and composition, strong morphology-, density-, particles dispersion-electrodeposition parameters relationship, etc [1]. Recently, the use of surfactant is likely one of useful methods to modify particles growth on electrode surface [21,22]. As has been well-known, the surfactant may control the nature of the metal ions reduction that manifested by the

change in the reduction rate and the crystallization characteristic of electro-deposit on electrode [23]. By simply adjusting surfactant concentration, nano-particles with controlled size, density and dispersion can be obtained [24,25]. These certainly influence the quality of the electrodeposited in general. Therefore, owing to the simplicity of electro-deposition technique to grow nanosized particles, the effort to improve the growth characteristic of the Co-Ni-Cu nanoalloys using this method in order to produce excellent particles size distribution, dispersion and density should be continuously demonstrated [24].

In this study, the CoNiCu alloy nano-particles called nanoalloys were prepared by electrodeposition technique from the electrolyte containing alkyl polyglucoside (APG). APG is a glycolipid surfactant [26] that exhibits excellent physicochemical proper-ties and environmental compati-bility [27, 28]. To modify morphology and chemical compo-sition of the alloy, the electrodeposition potential was varied. The effect of the APG surfactant on morphology, chemical composition and structure of the alloy are discussed.

2. MATERIALS AND METHODS

The chemicals used for sample preparation were $CoSO_4.7H_2O$, $NiSO_4.6H_2O$, $CuSO_4.5H_2O$ and H_3BO_3 which were supplied by Sigma-Aldrich. Alkyl polyglucoside as the surfactant was purchased from Fluka. The indium-tin oxide coated glass substrate (ITO) with diameter of 1.1 cm was supplied by Praezisions Glas & Optik GmbH. The sheet resistance of ITO substrate is 10 ohms/square.

The electrochemical experiments were carried out in a three-electrode cell with the ITO substrate, a platinum wire and a saturated calomel electrode (SCE) acted as working electrode, counter electrode and reference electrode, respectively. The ITO substrate was firstly cleaned using ultrasonic cleaner in acetone and then rinsed by a copious amount of de-ionized water. The cyclic voltammetry measure-ments were conducted in an electrolyte containing $CoSO_4$, $NiSO_4$, $CuSO_4$ of 0.20 M and H_3BO_3 of 0.4 M. The CoNiCu nanoparticles were prepared from the electrolyte by potentio-static technique performed using a GAMRY PCI4 potentiostat that controlled with DC105 software. In the preparation process, we studied the Co-Ni-Cu nanoalloys electro-deposition in the absence and in the presence of 3.25 wt.% of alkyl polyglucoside. In typical procedure, a pure argon gas flow was purged into the electrolyte for 15 minutes to remove any oxygen content before performing the sample preparation.

Structural characterization of the sample was carried out by a Bruker D8 Advance X-ray diffractometer (XRD) with grazing incidence configuration (GIXRD). Morphological analysis was examined using a SUPRA 55VP field emission scanning electron microscope (FESEM). The elemental mapping and chemicals composition of the deposits were examined using an Oxford energy dispersive X-ray spectrometer (EDX) which is attached to the FESEM.

3. RESULTS AND DISCUSSION

Figure 1 shows potentiostatic current transients for electrodeposition of cobalt, nickel and copper at co-deposition potential of -950 mV vs SCE for 150 seconds, both in the absence and in the presence of the surfactant. Without APG, the nucleation was found to occur in the system as shown by the significant increase of the current at the first 20 seconds that followed by a diffusion process as indicated by a drastic decrease in the current density after reaching the maximum value [29]. From the curve, it can also be understood that the growth and the stability characteristics of the nuclei are excellent of which indicated by presence of

a steady linear increase in current density of the system up to a maximum value [30]. In contrast to the system without APG, the current transient obtained from the electrolyte that contains APG indicated a slower nuclei formation. This could be probably due to a successful adsorption of the surfactant molecules onto the ITO surface that covered the active area of the substrate [24,31]. This in turn may result in the inhibition of ions transfer from the electrolyte onto the electrode surface [32] and certainly decreased the metal ions reduction on the surface. Hence, a low current density was recorded from the electrolyte containing the surfactant.

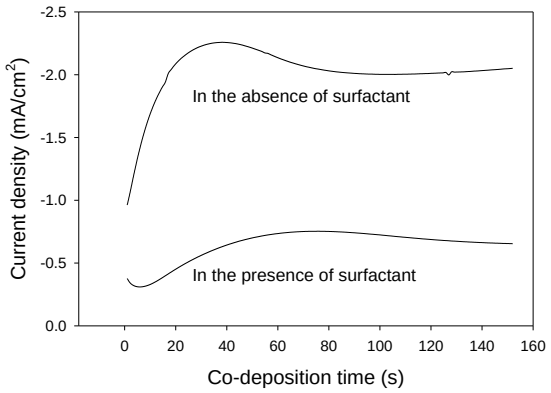

Figure 1. *Potentiostatic current transients recorded in the electrolytes containing 0.2 M of Co^{2+}, Ni^{2+} and Cu^{2+} both in the absence and in the presence of alkyl polyglucoside.*

FESEM micrograph of the Co-Ni-Cu nanoparticles prepared with with different concentration of surfactant are shown in Figure 2. The micrograph shows that the Co-Ni-Cu nanoalloys prepared without the surfactant exhibits the formation of irregular shape nanoparticles that agglomerated and formed a dense deposit (Figure 2a). However, characteristics of the CoNiCu particle found to be different when the APG surfactant was introduced into the electrolyte. In this case, the particles shape was much uniform compared to those without surfactant, namely spherical with size of approximately 20-50 nm. Interestingly, no particles agglomeration was found on the surface (Figure 2b). This result could be attributed to the surfactant adsorption on the particles surface that protected them from agglomeration with other particles [24].

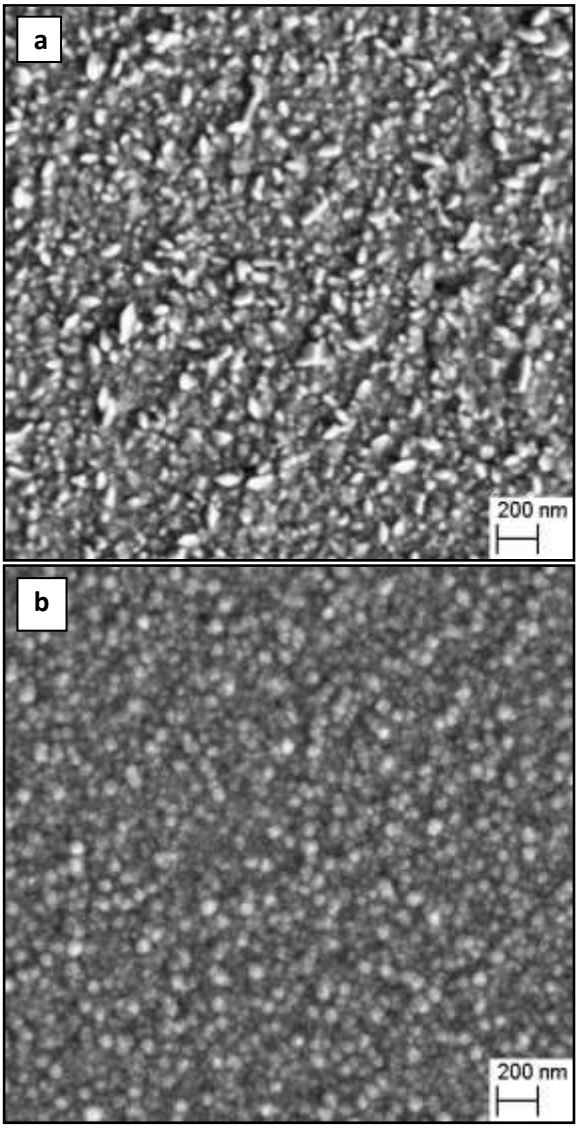

Figure 2. *FESEM micrograph of Co-Ni-Cu nanoalloys prepared at potential of -950 mV vs SCE (a) in the absence and (b) in the presence of APG surfactant.*

Moreover, the adsorption of the surfactant on the substrate could provide a template

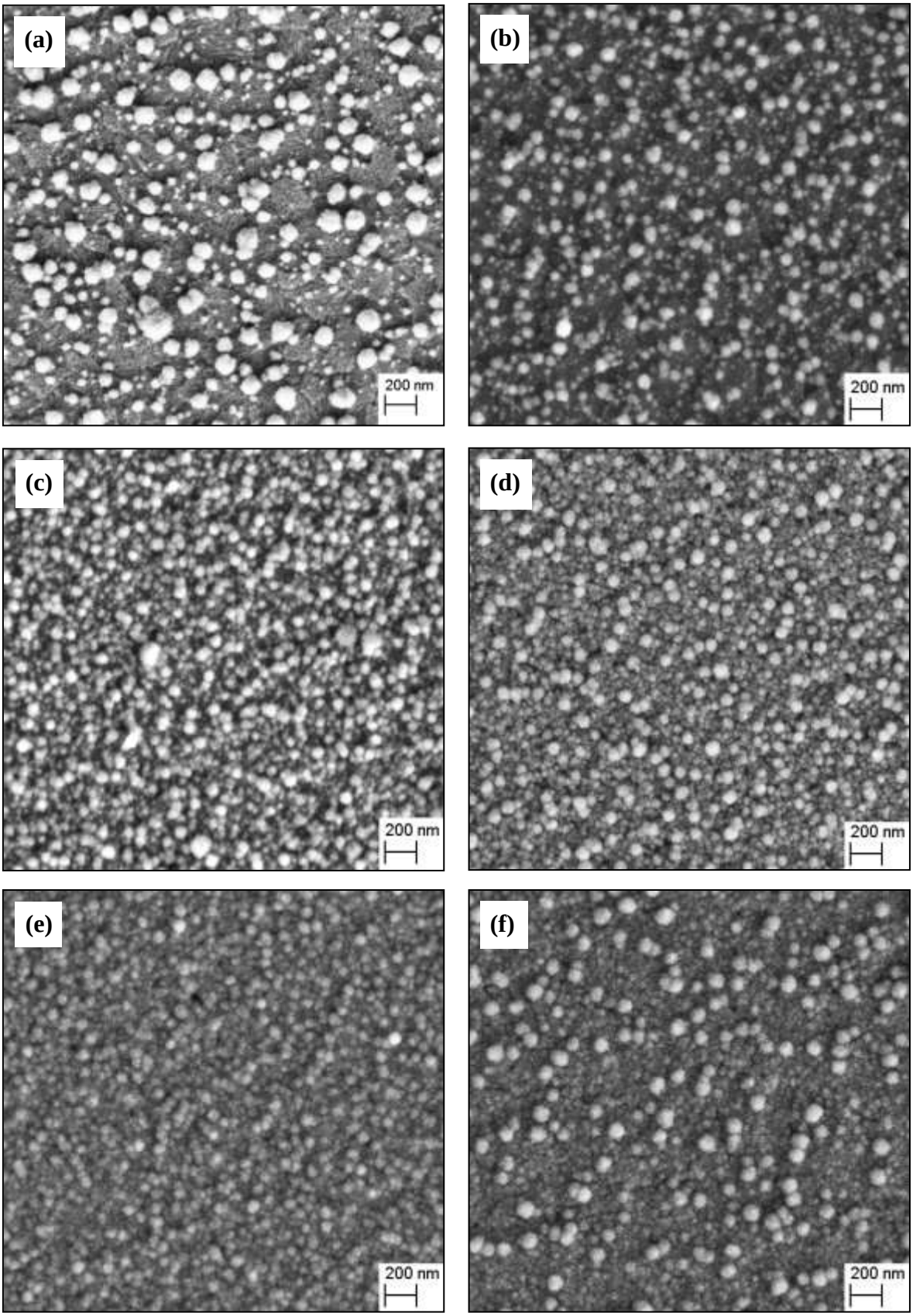

Figure 3. *FESEM micrographs of Co-Ni-Cu nanoalloys particles obtained from electrolyte containing APG surfactant at different electrodeposition potential (a) -850, (b) -875, (c) -900, (d) 925, (e) -950 and (f) -975 mV vs SCE.*

[33] that modified the particle growth characteristic that can be assumed to play a role in the formation of spherical shape and well-dispersion nanoparticles on the surface. The present result is in good agreement with the previous study [34] which reported such benefit of surfactant in controlling the shape and growth characteristic of the electrodeposited nanoparticles.

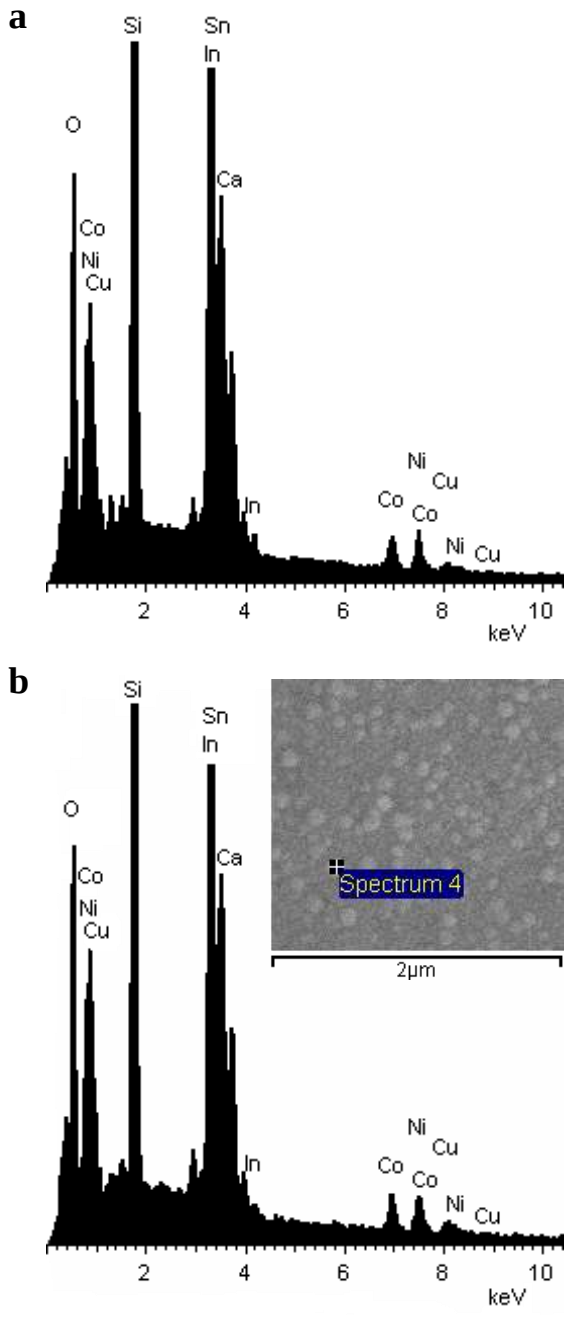

Figure 4. *(a) EDX spectrum obtained from Co-Ni-Cu nanoalloys prepared at electrodeposition potential of -925 mv vs SCE, (b) EDX point analysis.*

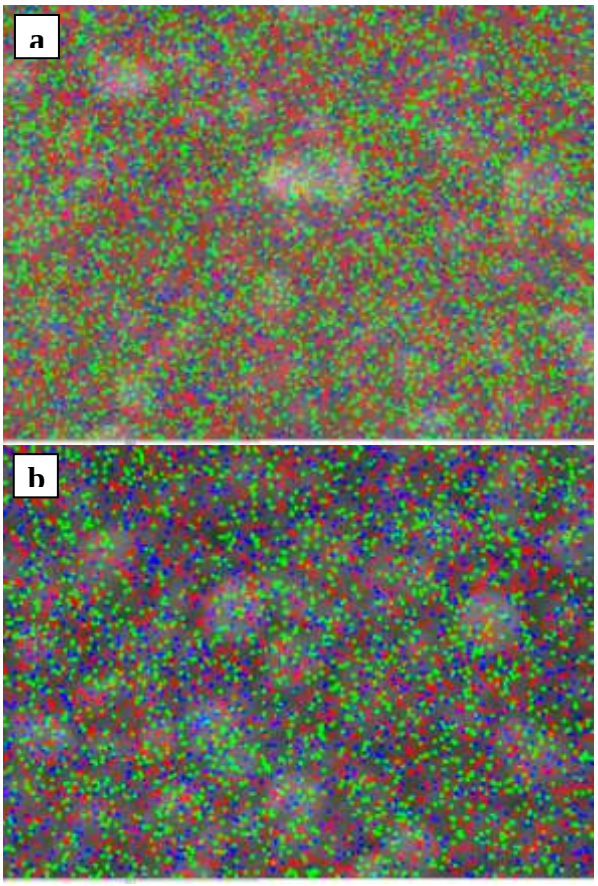

Figure 5. *Elemental mapping of cobalt, nickel and copper electrodeposited at potential of -925 mV vs SCE (a) in the absence and (b) in the presence of APG surfactant.*

To further understand the particle growth characteristic in the presence of APG, the CoNiCu electrodeposition were performed at different deposition potential, namely from -850 to -975 mV vs SCE. The results are shown in Figure 3. The FESEM micrographs show that the nanoparticle characteristics, such as morphology, density and dispersion, indicated the strong dependency on the applied deposition potential. For example, at lower potential, -850 mV vs SCE, scarce and bigger particles growth on the surface was obtained from the system. However, the size of particles significantly decreased when the applied potential increased. Meanwhile, for the case of density, the nanoalloys particles density underwent a significant increase with the enhancement of applied deposition potential. At high

potential, deposits that composed by high density nanoparticles were obtained. Based on this result, in the presence of APG, the applied potentials also played a key function in controlling the deposit characteristic of the Co-Ni-Cu nanoalloys.

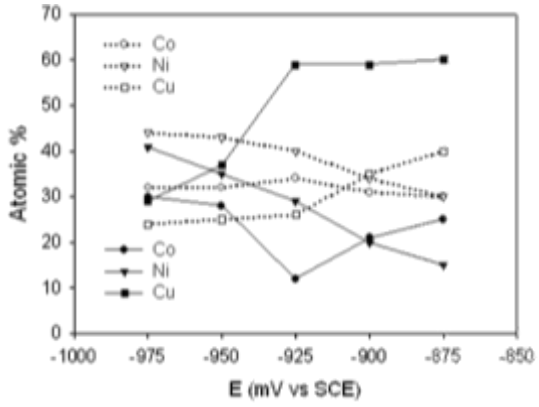

Figure 6. *Composition of cobalt, nickel and copper in deposited films in the absence (.....) and in the presence (——) of APG surfactant.*

Figure 4 shows EDX spectra that confirm the existence of cobalt, nickel and copper. Other signals come from some elements of ITO substrate and glass plate since the Co-Ni-Cu deposits formed on the substrate is quite thin. The point analysis was also performed on a particle of the same sample to confirm the CoNiCu alloy formation. The result shown in Figure 4b indicated the existence of the three elements in the prepared nanoparticle. To further confirm the element distribution in the particles', elemental mapping analysis was carried out using EDX. Figure 5 presents the distribution of cobalt, nickel and copper which are indicated with the red, green and blue dots, respectively. It is found that all the three metal components were found to be homogenously distributed throughout the electrodeposited nanoparticles, reflecting the formation of CoNiCu alloy.

Figure 6 shows the average compositions of the CoNiCu alloy which were prepared at different potentials in the absence and the presence of the APG surfactant. For the results obtained without surfactant, there was no significant change observed on the cobalt content if the reduction potential increased. While in the presence of surfactant, the content of nickel was found to increase, while copper decreased with the increasing of the applied potential. This could be associated to increase of co-deposition overpotential in the presence of surfactant (Q. Wang, Wang, Wang, & Xu, 2019) that reduced the deposition rate of the copper which has lower reduction potential compared to that of nickel and cobalt.

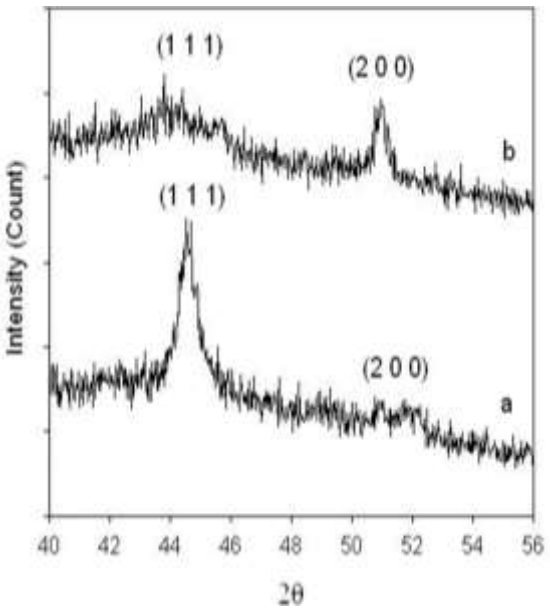

Figure 7. *GIXRD patterns Co-Ni-Cu nanoalloys deposits obtained (a) in the absence and (b) in presence of APG surfactant.*

To further confirm the formation of CoNiCu alloy, the samples were analyzed using grazing incidence X-ray diffractometer technique. The XRD patterns are shown in Figure 7. For the case of the sample prepared in the absence of APG, the peaks at 2Θ 44.53° and 51.62° were observed. These peaks could be assigned to the (1 1 1) and (2 0 0) planes of CoNiCu alloy structure [36–38], respectively. In the presence of surfactant, the pattern exhibits a strong peak at (2 0 0) instead of (1 1 1) plane. This certainly reflected a prefer orientation of the (2 0 0). This result implies that the APG surfactant

not only modified the composition of the alloy, but also altered the crystal growth orientation.

At this moment, exact function of APG in modifying the deposition characteristic of the Co-Ni-Cu nanoalloys, such as morphology, composition, etc, is not yet well-understood. However, the following assumptions can be considered: firstly, the APG might effectively attach onto the crystallite deposits and control the growth orientation of the nanoalloys particles. The {1 0 0} crystallographic planes are likely the preferred surface for the APG to be adsorbed. It is indicated by the strong X-ray diffraction by this plane as shown in the XRD pattern. Secondly, the APG attached onto the nanoparticles surface is also effectively control the nucleation process of the metallic atoms onto specific crystallographic plane. This is resulted in the slowing the nanocrystal growth. Consequently, large numbers of new crystallites were formed and in turn produced a high density smaller nanoalloys particle. Thirdly, the APG surfactant adsorption on the electrode surface was also occurred on the metal ions species. This may cause the increased in the overpotential of the electrodeposition process. As the results, the chemical composition of the nanoalloys particles will be modified compared to those obtained without APG. The formation of template by the APG surfactant can also be related to the formation of unique shape of the nanoparticles on the surface.

4. CONCLUSION

The Co-Ni-Cu nanoalloys were successfully prepared using the electrodeposition technique. In the presence of APG, the nanoparticles with excellent size homogeneity and particles density could be obtained. The APG was also found to modify the crystal preferred growth orientation as shown by the XRD results from {1 1 1} of the system without APG to {2 0 0} of the system with APG. It was also found that APG also modified the chemicals composition of the nanoalloys. The modification of the crystal growth orientation of the nanoalloys was considered as successful adsorption of APG molecules on the crystal surface as well as metallic ion species. Thus modify the growth characteristic.

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CONFLICT OF INTEREST

The author declares that there is no conflict of interest regarding this publication.

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