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Structural and morphological properties of NiCu magnetic thin films

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NiCu thin films for magnetic applications have been produced by using an electrochemical method. Their structural and morphological properties have been investigated by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM and EDX) and are discussed in correlation with the growth conditions.

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Keywords: NiCu, XRD, SEM, EDX

1. Introduction

Due to their magnetic and magnetoresistive properties, metallic alloy films, which are composed of magnetic-nonmagnetic or magnetic-magnetic elements, have attracted a great deal of interest in the past decade. Systems such as NiCu [1], FeCu [2], and CoCu [3] are good examples of magnetic-nonmagnetic magnetoresistive systems. Nickel-based alloys are particularly attractive because of their magnetic properties. The magnetic response of thin layers based on nickel alloys, always exhibits a strong dependency of composition, size, shape and morphology. The films were prepared by different methods, among them the most used being vacuum evaporation, electrodeposition and sputtering on nonmagnetic substrate. Electrodeposition, which is a relatively low-cost technique, does not need any sophisticated equipment and may be easily used to grow NiCu films with different properties by controlling the growth parameters.

In this paper we report on the results of an investigation on the structural and morphological properties of NiCu magnetic thin films, to identify the influence of growth parameters on the quality of the films. Such a study is justified by the potential applications of NiCu thin films for magnetic field sensors [4], giant magnetoresistance systems, in magnetic recording as MRAM, HDD etc.

2. Experimental results and discussion

NiCu magnetic thin films were obtained by using an electrodeposition method. The films were electrodeposited in a Watts bath. The composition of the basic plating solution was: nickel sulphate hexahydrate (225 g/l), nickel chloride hexahydrate (30 g/l), boric acid (22,5 g/l) and copper sulphate pentahydrate (4 g/l). An organic additive (PVP – polyvinyl pyrrolidone, 5 g/l) was used as wetting agent.

As a first step of the deposition procedure, a gold thin film, 50 nm thick, was sputtered on the surface of the glass substrate. The gold film was subsequently used as working electrode during the deposition of the magnetic alloy. The electrochemical deposition was performed by using a VoltaLab potentiostat controlled by a computer. The temperature was kept constant, at 65º C. A three electrode configuration was used, with a platinum counterelectrode and a commercial saturated calomel electrode (SCE) as reference. During the deposition, the electrolytic bath was mechanical stirred for homogenization.

Three successive polarization curves, recorded during deposition, are shown in Fig. 1.

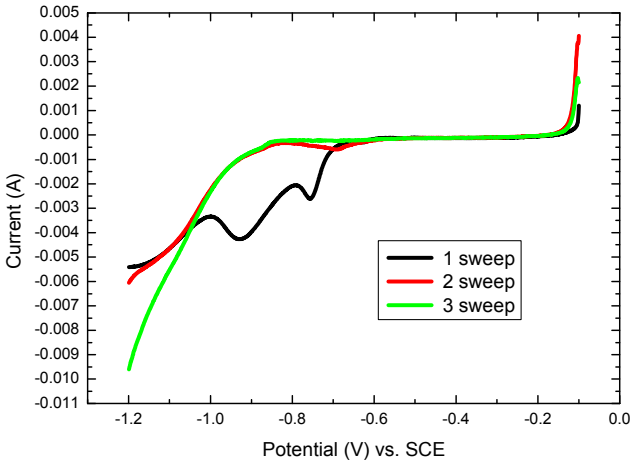


Fig. 1. Voltammetric curves recorded during the deposition of NiCu thin films, in the conditions specified in text.

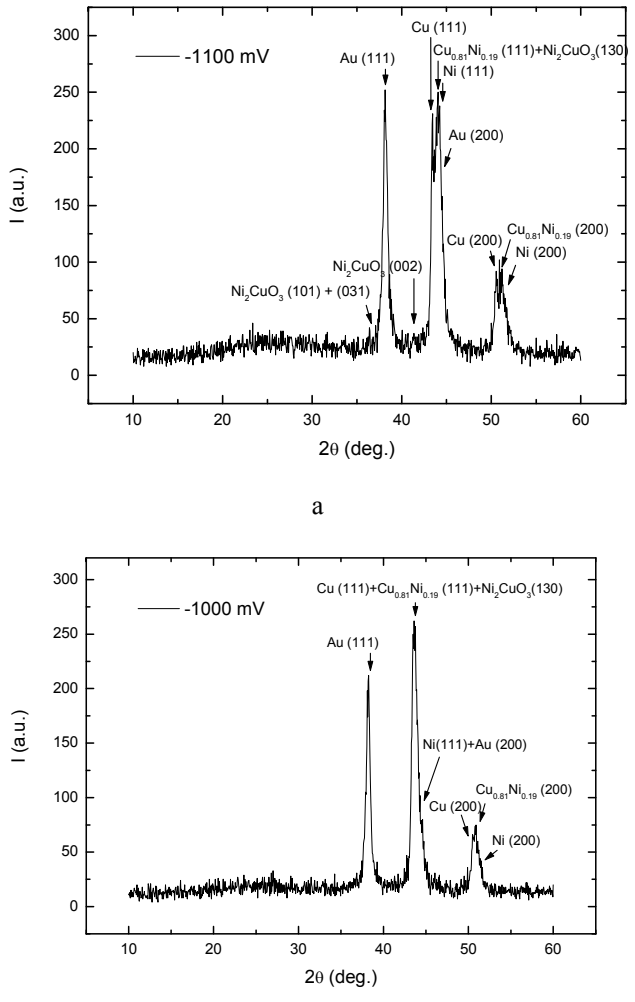
The flat region extending from -200 mV to -850 mV is associated with a stoichiometric compound deposition, while the region from -850 mV to -1200 mV is associated with the prevalent deposition of the element which has a higher concentration in the bath (Ni + NiCu). There are not significant differences between the second and the third runs, hence the deposition process does not depend essentially on the substrate material.

The crystallinity of the films was characterized by X-ray diffraction (XRD), using a high resolution X-ray diffractometer (D8 Discover – Bruker). The films morphology and composition were investigated by SEM, respectively EDX techniques.

XRD spectra were recorded by using Cu-$K_{\alpha 1}$ line, λ = 1.5406 Å, in a grazing incidence geometry for increasing the path length of the X-ray beam through the film. XRD patterns are shown in Fig. 2.

A richer structure of the diffraction peaks can be seen in the case of the sample electrodeposited at -1100 mV vs. SCE (see figure 2a). There is evidence for the presence of a $Cu_{0.81}Ni_{0.19}$ phase in the analyzed film. Also the peaks due to reflections on (111) planes of Au, Cu and Ni separate phases can be clearly seen. In the case of the sample deposited at -1000 mV vs. SCE (shown in figure 2b), the diffraction peaks are featureless, although suggesting the presence of the same crystalline phases, with an increased content of Cu, as a separate phase.

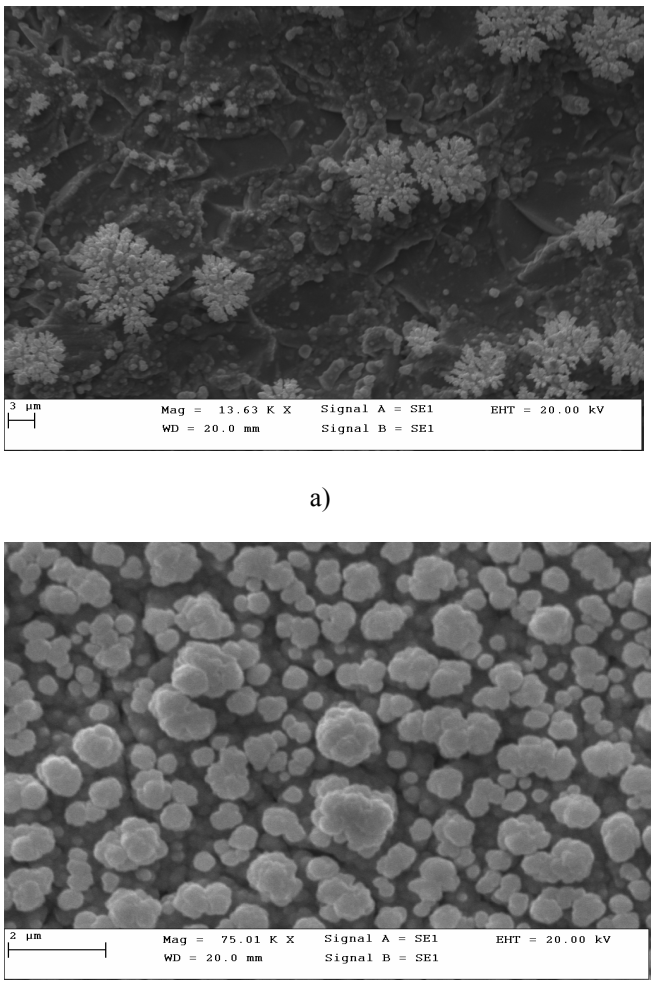
Scanning electron microscope (SEM) images of films deposited at different potentials are shown in figure 3, while figures 4 and 5 show the results of a composition analysis of the films, as obtained using Energy Dispersive X-ray Spectroscopy (EDX).



a

b

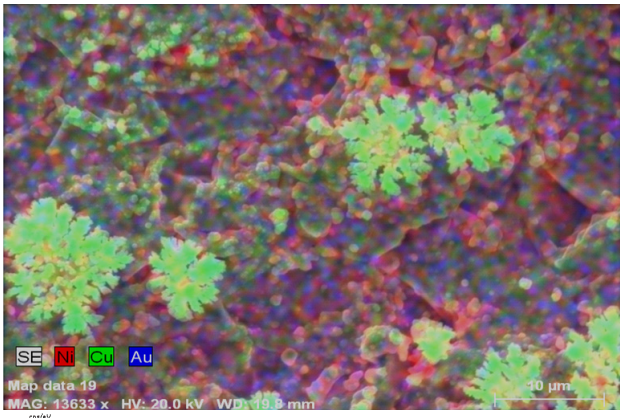
Fig. 2. Experimental X-ray diffraction pattern of analyzed NiCu samples.

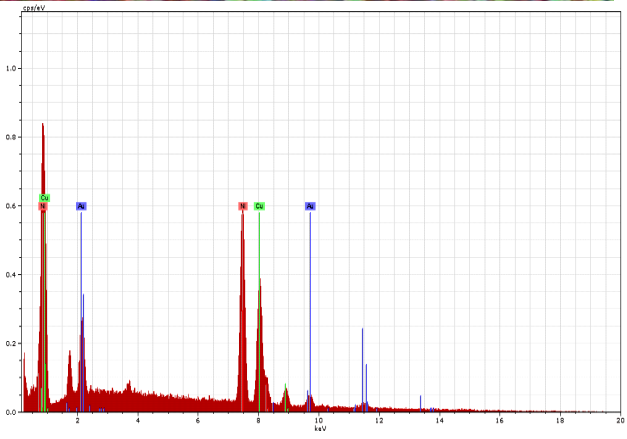


a)

b)

Fig. 3. SEM micrographs of NiCu samples deposited at -1100 mV vs. SCE (a) and, respectively, at -1000 mV vs. SCE (b).



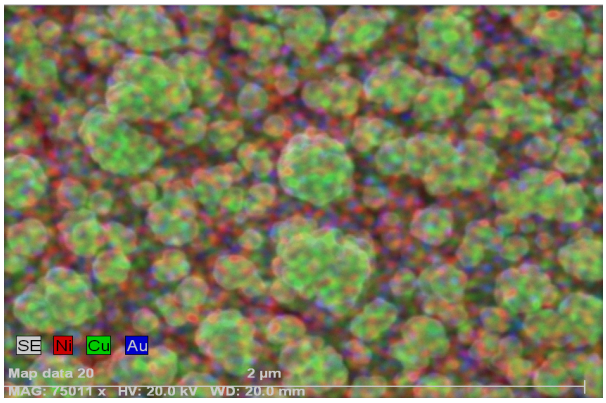


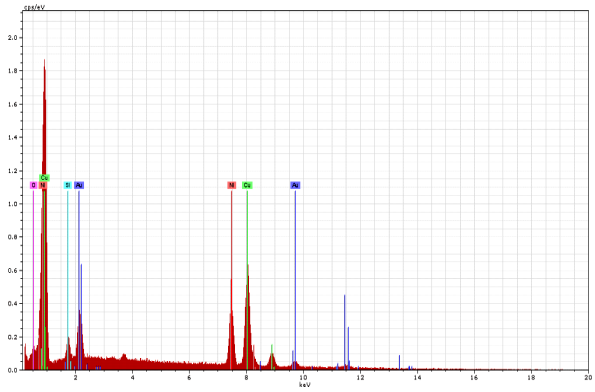
Spectrum: Acquisition

El	AN	Series	Net	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at.-%]	Error [%]
Ni	28	K-series	36344	38.14	44.20	52.39	1.1
Cu	29	K-series	24076	32.47	37.63	41.20	0.9
Au	79	L-series	3746	15.67	18.17	6.42	0.6
		Total:		86.28	100.00	100.00	

Fig. 4 SEM-EDX elemental mapping, spectrum and elemental composition of sample grown at -1100 mV vs. SCE.

The films grown at -1100 mV showed an overall composition of 52.39 at.% Ni and 41.20 at.% Cu, while for the film grown at -1000 mV an overall composition of 21.97 at.% Ni and 45.79 at.% Cu was detected. Clearly, more negative potentials favor Ni deposition and also the forming of $Cu_{0.81}Ni_{0.19}$ phase. The films deposited at -1100 mV vs. SCE are also more compact, with larger crystalline grains.





Spectrum: Acquisition

El	AN	Series	Net	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at.-%]	Error [%]
Si	14	K-series	3520	1.89	2.00	4.15	0.3
Ni	28	K-series	16689	20.86	22.12	21.97	0.6
Cu	29	K-series	28632	47.07	49.92	45.79	1.3
Au	79	L-series	3673	18.72	19.86	5.88	0.7
O	8	K-series	4625	5.75	6.10	22.22	0.9
		Total:		94.29	100.00	100.00	

Fig. 5. SEM-EDX elemental mapping, spectrum and elemental composition of sample grown at -1000 mV vs. SCE

3. Conclusions

Using an electrodeposition method, NiCu magnetic thin films were prepared. An investigation on the effect of growth parameters on the quality of the layers was performed. SEM and XRD were used to investigate the morphology and crystallinity of obtained structures, respectivele. The XRD analysis revealed that all NiCu thin films were polycrystalline. Electrodeposition at more negative potentials results in films richer in Ni, present either in a $Cu_{0.81}Ni_{0.19}$ crystalline phase or as a separate elemental phase.

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