Quantitative SEM study of NiCo alloy powders electrodeposited on Cu substrates

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Nanostructured metals and alloys have an attractive potential for technological applications [1]. The electrodeposition technique has significant advantages over other methods for synthesizing nanocrystalline materials such as the potential of synthesizing a large variety of nanograined materials: pure metals, alloys and composite systems with grain sizes as small as 20 nm [2].

Nickel and cobalt alloy powder deposits from three different electrolyte compositions were obtained by electrodeposition from an ammonium sulfate-chloride solution in a galvanostatic regime. The effect of both current density and bath composition was studied by methods of scanning electron microscopy (SEM) and X-ray diffraction. It was found that the level of overpotential significantly affects the structure and composition of the formed alloy deposits. An increase of the volume fraction of the hexagonal-close packed (hcp) phase in the nanocrystalline deposit is caused by an increase of the Co^{2+} ion concentration in the bath and by a decrease of deposition current density; Whereas an increase of the current density and a decrease of the Co^{2+} ion concentration in the bath yields finer grain deposits. For a chosen current density of $65 mAcm^{-2}$ particles of different morphology (with sizes from 5 to 50 μm) composed from fine nanosized crystallites are obtained on Cu substrates (Fig. 1). When $Ni^{2+}:Co^{2+}=4:1$ in the electrolyte, the deposit has a cauliflower structure surrounded with diffusion zones as shown in Fig. 1a; the average grain size is about 13 nm as determined by X-ray diffraction methods. In contrast, the particles deposited from the electrolyte with $Ni^{2+}:Co^{2+}=1:1$ and $Ni^{2+}:Co^{2+}=1:4$ show platelet structures with preferred orientations (cf. Fig. 1b and 1c). The sizes of the platelets are in μm range and the grain sizes are in the range from 15 to 20 nm.

The 3D reconstruction of the specimen surface shown in Fig.2 was characterized by SEM using MeX software from Alicona**.** It enables to carry out a 3D analysis directly from the digital images yielding profile and roughness measurements and also area analysis as well as volumetric measurements (cf. Table 1). Surface morphology and roughness of the deposits depend on the concentration ratio in electrolyte. In the case of $Ni^{2+}: Co^{2+} = 1:1$ the roughness of the deposit is almost 3 times higher than in the specimens with ratios of 4:1 or 1:4. Also, the active surface has a maximum at the ratio 1:1. An increase in the current density results in a decrease in roughness, since at higher current densities the number of crystal nuclei on the surface in enhanced. The larger the nucleation rate, the smaller is the grain size, leading to the formation of smooth deposits when the grain size ≤ 10 nm.

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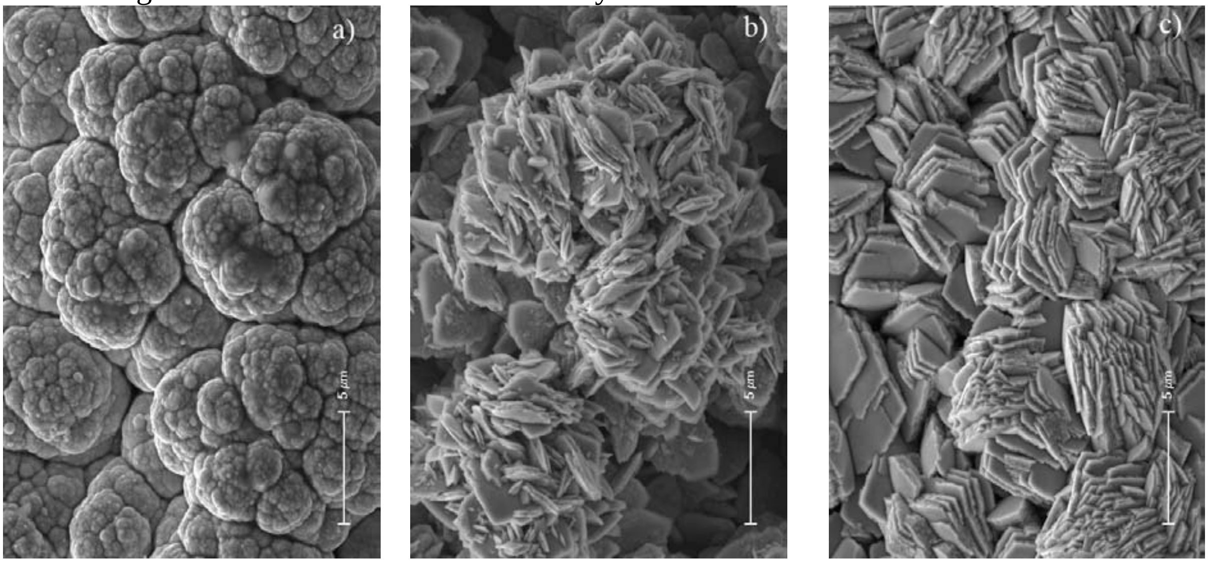

Figure 1. SEM micrographs of Ni-Co deposits obtained in the galvanostatic regime at a current density 65 mAcm^{-2}. The concentration ratios Ni^{2+}: Co^{2+} in the electrolyte were: 4:1 in a), 1:1 in b) and 1:4 in c).

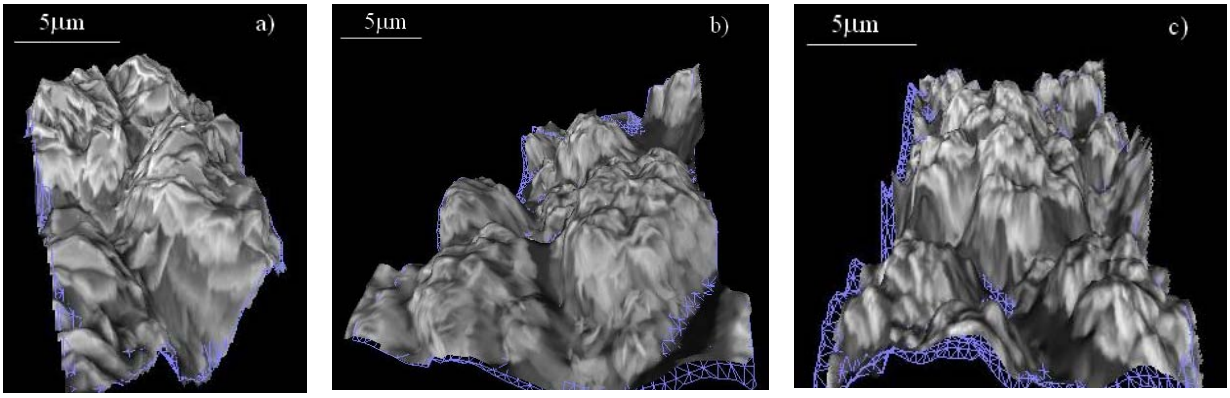

Figure 2. 3D SEM reconstructions of the surfaces of Ni-Co deposits from an electrolyte with concentration ratio Ni^{2+}:Co^{2+} =1:1 at different current densities: a) 65, b) 220 and c) 400 mAcm^{-2}. (Fig.2a) corresponds to Fig. 1b)).

Ni^{2+}:Co^{2+} concentration ratio	current density (mAcm^{-2})	Ra (µm)	Rz (µm)	RS (µm^2)
4:1	65	1.0	4.7	1.64
1:1	65	3.0	13.1	1.98
1:1	220	1.1	5.2	1.38
1:1	400	1.0	6.5	1.62
1:4	65	0.7	4.7	1.64

Table 1. Evaluation of the quantitative SEM results. Roughness parameters of alloys deposited from electrolytes with different Ni^{2+}:Co^{2+} concentration ratios and current densities (Ra: mean roughness; Rz :the difference between the highest and the lowest point in the picture of a given scan; RS :active surface, ratio of the real surface including topography to a projected surface of the measurements in a square with dimensions of 23x15 µm).

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