# ELECTROCHEMICAL DEPOSITION OF POWDER OF TERNARY Co-Ni- Mo ALLOY FROM ALKALINE ELECTROLYTE

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# ABSTRACT

The Co-Ni-Mo alloy powders were electrochemically deposited from alkaline ammonical electrolyte containing two or three different elements, Co, Ni and/or Mo at a constant current density. The obtained Co-Ni-M powders were studied using EDS, X-Ray and SEM analysis. Based on the obtained experimental results it can be concluded that by appropriate selection of chemical composition of the electrolyte for deposition, varying of Co, Ni and Mo ions ratios and current density it can be influenced on the particle sizes of deposited powders. X-ray results suggest that the obtained powders have amorphous structure.

Keywords: Co-Ni-Mo alloys, alkaline electrolyte, electrochemical deposition

## **1. INTRODUCTION**

Electrochemically deposited iron-group metals alloys, whether in a form of powders or coatings are very important magnetic materials [1-3], as well as good catalysts for hydrogen evolution [4]. Powders of binary and multicomponent alloys and metal deposits are divided according to the type of deposition that they undergo, in a system defined by Brenner [5].

### 2. EXPERIMENTAL

The powders were electrodeposited from the electrolyte containing:  $CoSO_4$ ,  $NiSO_4$ ,  $(NH_4)_6Mo_7O_{24}$ ,  $NH_4Cl$  and  $NH_4OH$ . Different  $Ni^{2+}/Co^{2+}$  concentration ratios were used  $Ni^{2+}/Co^{2+}$  (1:1) [0.1M Ni / 0.1 M Co] and (4:1) [0.16 M Ni / 0.04 M Co]. The molybdate concentration of was also varied from 0.005 to 0.06 mol/dm<sup>3</sup> and the NH<sub>4</sub>Cl concentration was 74.13 g/dm<sup>3</sup>. The NH<sub>4</sub>OH was added to keep the pH of the applied solutions constant at 10.6

The morphology and the particle size of electrodeposited powders was observed using scanning electron microscope (SEM) JEOL JSM-6460 LV.

The EDS analysis carried out on the Oxford Instruments-INCA Energy system, provided the quantitative chemical composition of the deposited Co-Ni-Mo alloy powders.

Phase compositions of the obtained powders ware determined by the X-ray diffraction analysis performed on an X'Pert PRO MPD multi-purpose X-ray diffraction system from PAN analytical. Data were collected in a step scan mode between  $20^{\circ}$  and  $110^{\circ} 2\theta$  at every  $0.02^{\circ} 2\theta$ .

#### **3. RESULTS AND DISCUSSION**

The results of the EDS analysis presented in tables (1, 2) indicate that Ni content in the deposit is practically independent of current density and Ni concentration in the bath for deposition. With an increase of the Ni<sup>2+</sup>/Co<sup>2+</sup> concentrations ratio to (4:1) Ni content in the deposit is increased only for one or two percent. Therefore, it is obvious that Co preferentially induces Mo deposition and that the influence of Ni is almost negligible.

(N1 / CO (1.1), 400  mA/cm)				
Mo concentration in the electrolyte (mol/l)	O (at%)	Co (at%)	Mo (at%)	Ni (at%)
0.005	72	11	14	3
0.01	62	15	30	3
0.03	44	18	36	2
0.06	44	18	36	2

Table 1. The chemical composition of electrodeposited Co-Ni-Mo alloy powders  $(Ni^{2+}/Co^{2+} (1:1), 400 \text{ mA/cm}^2)$ 

Table 2. The chemical composition of electrodeposited Co-Ni-Mo alloy powders  $(Ni^{2+}/Co^{2+} (1:1), 800 \text{ mA/cm}^2)$ 

Mo concentration in the electrolyte (mol/l)	O (at%)	Co (at%)	Mo (at%)	Ni (at%)
0.005	63	12	22	3
0.01	50	16	32	2
0.03	42	19	37	2

In the Co-Ni-Mo alloy powder, Mo can be present in a form of pure metal, oxide (MoO,  $Mo_2O_3$ ,  $MoO_2$ ,  $MoO_3$  and  $Mo_2O_5$ ) or even in a form compound with Co or Ni.

The presented results of EDS analysis of the deposited alloy powders suggest that every increase of the Mo content in the deposit is accompanied by an increase of the Co content and that it is almost impossible to influence on the Ni content in the deposit in spite of variation of  $Ni^{2+}/Co^{2+}$  concentration ratio in the electrolyte for deposition.

From the SEM micrographs of the deposited Co-Ni-Mo alloy powders presented on Fig.1 and Fig.2 it can be seen that structure consisting of very fine nanoscale particles lumped together in much larger agglomerates was formed.

At the (1:1)  $Ni^{2+}/Co^{2+}$  concentration ratio in the electrolyte and at the constant current density of 400 mA/cm<sup>2</sup> (Fig. 1a) particles having sizes 80-150 nm were deposited as at current density of 800 mA/cm<sup>2</sup> (Fig. 1c) the obtained particle sizes were in range of 50-70 nm.

Fig. 1a and Fig. 1c clearly illustrate the decrease of the particle size with the increase of a current density.

The alloy powder deposited from the electrolyte containing  $Ni^{2+}/Co^{2+}$  (1:1) concentration ratio [0.1M Ni, 0.1 M Co] and 0.03 M Mo at the current density of 800 mA/cm<sup>2</sup> consists of particles having sizes about 150 nm (Fig. 1b), whereas at the current density of 400 mA/cm<sup>2</sup> and twice as high Mo concentration (0.06 M) in the electrolyte smaller particle sizes are obtained (Fig. 1a). Much finer particles, even two to three times smaller are obtained with the increase of the Mo concentration in the electrolyte and at the constant current density of 800 mA/cm<sup>2</sup> (Fig. 1b, Fig.1c), indicating that by increasing of Mo concentration in the electrolyte and current density it can be influenced on the particle sizes.

On the SEM micrographs (Fig. 2) of the alloy powders deposited from the electrolytes with increased  $Ni^{2+}/Co^{2+}$  concentration ratio (4:1) and Mo concentration range 0.005 to 0.03 M at the constant current density of 400 mA/cm<sup>2</sup>, the decrease of the particle sizes in the deposit can be observed with the increase of the Mo concentration in the electrolyte.

By comparing the Fig.1 and Fig.2 the changes of the morphology of the deposited powders with the increase of the Ni in the electrolyte can be observed. The powders deposited from the electrolyte

containing (1:1)  $Ni^{2+}/Co^{2+}$  concentration ratio (Fig. 1) have primarily flake like particles of angular shapes while the powders deposited from electrolyte containing (4:1)  $Ni^{2+}/Co^{2+}$  concentration ratio (Fig. 2) are in the form of large agglomerated particles having more or less dense cauliflower structure.



a) 400 mA/cm2, (0.06 M) Mo

b) 800 mA/cm2, (0.03 M) Mo

c) 800 mA/cm2, (0.06 M) Mo

Figure 1. SEM micrographs of Co-Ni-Mo-O alloy powders, electrodeposited from the solution containing  $Ni^{2+}/Co^{2+}$  (1:1) concentration ratio [0.1M Ni, 0.1 M Co]



Figure 2. SEM micrographs of Co-Ni-Mo-O alloy powders, electrodeposited at the current density of  $400 \text{ mA/cm}^2$  from the solution containing Ni<sup>2+</sup>/Co<sup>2+</sup> (4:1) concentration ratio [0.16M Ni, 0.04 M Co]

The phase composition of the deposited Co-Ni-Mo alloy powders was studied by the X-Ray analysis. The X-Ray diffractograms of the Co-Ni-Mo alloy powders obtained at all investigated deposition current densities and from all investigated plating baths (different Mo concentration) indicate the amorphous structure.

### 4. CONCLUSION

It can be concluded that increase of Co and Mo concentrations in the electrolyte as well as increase of the current density affects the particle size in the deposit i.e. smaller particles are obtained. The increase of the Ni concentration in the electrolyte for deposition was found to change the morphology of the Co-Ni-Mo powders from primarily flake lake angular particles to agglomerated particles having cauliflower structure and to lower Co and Mo content in the deposit. Electrochemical deposition of ternary Co-Ni-Mo alloy powders is accompanied by the simultaneous hydrogen evolution, which is in fact the primary reaction. Obtained experimental results indicate that for the reaction of powder deposition the current efficiency increases with the decrease of current density.

## **5. ACKNOWLEDGEMENT**

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