Influence of glycine on the electrochemical deposition of Sn-Co alloy from gluconate electrolyte

T. Valkova[*], I. Krastev

Institute of Physical Chemistry, Bulgarian Academy of Sciences, 1113 Sofia, Bulgaria

The influence of glycine on the electrochemical processes of deposition and dissolution of tin, cobalt and tin-cobalt alloy from a gluconate/sulfate electrolytes are studied by means of cyclic voltammetry.

The addition of glycine to the weak acid electrolyte for deposition of *Sn-Co* alloy has no significant influence on the deposition of tin and relatively strong influence on the deposition of cobalt, as well as a strong effect on the deposition of Sn-Co alloy. The addition of glycine and the increase of its concentration lead to increase in the cobalt percentage in the coatings.

Dull coatings with high cobalt content can be deposited from the investigated electrolyte. Depending on the pH and glycine addition the deposition of coatings with cobalt content in the range from 0 up to 65 wt. % is possible.

Key words: electrodeposition, tin-cobalt alloys, glycine

INTRODUCTION

Tin-cobalt electrodeposited alloy coatings are extensively applied as a convenient and economic way to achieve an attractive finish on lock and door hardware, plumbing fixtures, tubular furniture, and automobile interior trim and fittings. In addition, bright tin-cobalt alloy coatings have mechanical and electrochemical properties similar to those of chromium coatings. However, chromium plating is hazard. In view of that, electrolytic tin-cobalt alloy coatings can be considered to effectively replace chromium coatings [1, 2].

Tin-cobalt alloys plating could also be a substitute for cadmium in electronic application since the alloy resist oxidation and has a low contact resistance [3]. The Co-Sn electrolytes used are environmentally friendly, non-toxic, non-corrosive, and the electrodeposition process takes place at high energy efficiency. Tin-cobalt coatings can be used as a substitute of precious–metal coatings, avoiding the use of highly toxic cyanide electrolytes [4].

Much attention has been recently paid in the field of lithium batteries for the synthesis of new negative materials as alternatives to graphite materials. Among them, tin-based compounds have reached a particular attention. Tin-cobalt alloys find extensive application in this field, because it has been shown that the addition of Co gives the highest specific capacity [5], avoiding mechanical stress due to the Li-intercalation process [6].

Electrodeposited lead–based alloys have been used as overlays for plain bearing for many years. The toxicity of lead requires searching for lead-free alternatives. Tin-cobalt alloys [7] and compositionally modulated Sn-Co alloy multilayer coatings can be used as alternatives [8].

According to the phase diagram Sn-Co alloy may be composed of various intermetallic compounds depending on its metal content [9]. The preparation of heterogeneous coatings offers possibilities for investigation of the self-organization phenomena as observed during the deposition of other cobalt alloys – i.e. those with indium and antimony [10].

The deposition of the Sn-Co alloys is performed from sulfate/gluconate, citrate, fluoride and pyrophosphate electrolytes [11-13]. The sulfate/gluconate bath is highly prone to oxidation.

Glycine is known to stabilize both alkaline and acidic plating baths, it exhibits high buffering properties, which is important for stabilizing of pH on the electrode surface during electrodeposition [14]. Electrolytes containing glycine were used for electrodepositon of Co [15, 16] and some Co alloys [17-19].

The aim of this work is to study the influence of glycine on the electrochemical deposition of Sn-Co alloys.

EXPERIMENTAL

The composition of the electrolyte for deposition of Sn-Co alloy coatings is given in Table 1.

To whom all correspondence should be sent:
E-mail: tvalkova@ipc.bas.bg

Distilled water and *p.a.* grade reagents were used. The experiments were performed in a glass cell of 100 cm^{-3} at room temperature without stirring of the electrolyte. The working electrode (1 cm^2) and the two counter electrodes were made of platinum. A reference electrode Ag/AgCl with $E_{Ag/AgCl}$ = + 0.197 V against the hydrogen electrode was used. All potentials in the present study are given against this reference electrode. The sweep rate of the potential was 0.020 V s^{-1}.

Table 1. Electrolyte composition

| Composition	Concentration, g dm^{-3}
Sn as (CH$_3$SO$_3$)$_2$Sn	0-5
Co as CoSO$_4$.7H$_2$O	0-5
Na$_2$SO$_4$.	0-50
C$_6$H$_{11}$NaO$_7$	0-50
C$_2$H$_5$NO$_2$	0-50
KOH	0-5

The cyclic voltammetric investigations were performed by means of a computerized potentiostat/galvanostat PAR 273A (Princeton Applied Research) using the PowerCorr software for electrochemical corrosion studies.

The alloy coatings, *ca*. 5 µm thick, were deposited on 0.3 mm thick copper substrates, 2 x 1 cm in an electrolysis cell of 100 cm^3. The cobalt content in the coatings as well as their thickness was determined using a Fischerscope XRAY-XDAL apparatus for X-ray fluorescence.

RESULTS AND DISCUSSION

Tin deposition

Figure 1 shows the effect of the complex forming agent for tin (C$_6$H$_{11}$O$_7$Na in this case) on the electrode processes. The main function of sodium gluconate is to inhibit the hydrolysis of Sn(II) ions. Gluconate form soluble complexes in both slightly acidic and alkaline baths. Molar ratio of Na-gluconate to Sn should be at least 1.5 : 1 [8]. The presence of C$_6$H$_{11}$O$_7$Na in the electrolyte leads to a slight polarization and inhibition of the cathodic reaction. Gluconate is designated as GH$_4^-$, and the corresponding acid is gluconic acid, i.e. HGH$_4$, where the first H refers to the carboxylic acid hydrogen and H$_4$ refers to the four hydrogen
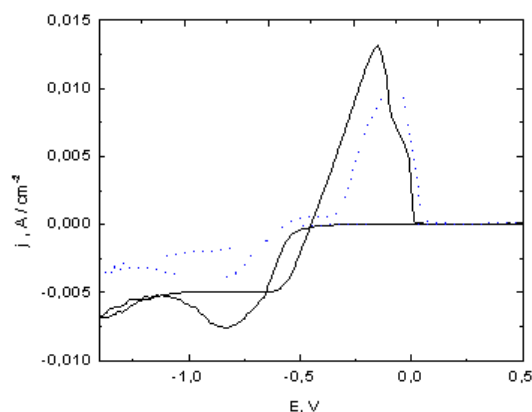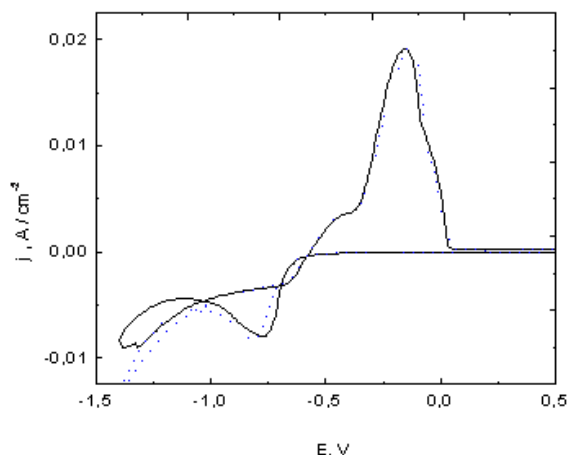

Fig.1. Influence of gluconate on the electrode processes during electrodeposition of tin from *sulphate electrolyte* at pH=3.5.C_{Sn}=5 g dm^{-3}; $C_{Na_2SO_4}$=40 g dm^{-3} —$C_{C_6H_{11}NaO_7}$=0 g dm^{-3}; ----$C_{C_6H_{11}NaO_7}$=50 g dm^{-3}; v= 20 mV s^{-1}

atoms on the secondary alcohol. Gluconic acid forms two kinds of complexes [20]. In acid media, the metal ion is bound into a complex via a carboxyl group ligand. The stability constants have relatively small values. Marksin et al. [21] reported that tin forms Sn(GH)$_4^+$ and Sn(GH$_4$)$_2$ complexes with stability constants logK = 3.01 and logK = 2.28. In presence of gluconate the tin reduction starts at more negative values, due to the formation of tin-gluconate complexes.

Two anodic reactions are detected at case of dissolution of tin in presence of gluconate ions instead of one observed in the absence of gluconate. The first oxidation peak is small and is related to the complexing action of gluconate ions on tin ions produced during oxidation process [22].

Figure 2 shows the cyclic voltammetric curves obtained for tin electrodeposition from sulphate/gluconate electrolyte with addition of 50 g dm^{-3} glycine. The presence of glycine in the gluconate containing electrolyte at pH=3.5 has very slight effect on deposition of tin (Fig. 2a).

At pH=5 (Fig 2b) during the deposition of tin two cathodic reactions are detected corresponding to deposition from different complexes. According to Survila [23] three complex species can be present in comparable amounts in such electrolyte. Sulphate complexes prevail in more acidic media and, as pH increase, they are replaced by Sn(GH$_4$)$_2$. Also Sn(OH)$^+$ and Sn(OH)$_2$ products of Sn(II) hydrolysis, can be formed at pH > 4, but this process is kinetically impeded [23]. The addition of glycine in the electrolyte has negligible effect on the first cathodic process but leads to some depolarization of the second cathodic reaction which correspond to the tin gluconate complexes in the presence of glycine (Fig. 2b).

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Fig. 2a. Influence of glycine on the electrode processes during electrodeposition of tin from *sulphate/gluconate electrolyte* at pH=3.5.C_{Sn}=5 g dm^{-3}; $C_{Na_2SO_4}$ =40 g dm^{-3};$C_{C_6H_{11}NaO_7}$= 50 g dm^{-3}; —$C_{C_2H_5NO_2}$ = 0 g dm^{-3}; ----$C_{C_2H_5NO_2}$ = 50 g dm^{-3}; v= 20 mV s^{-1}
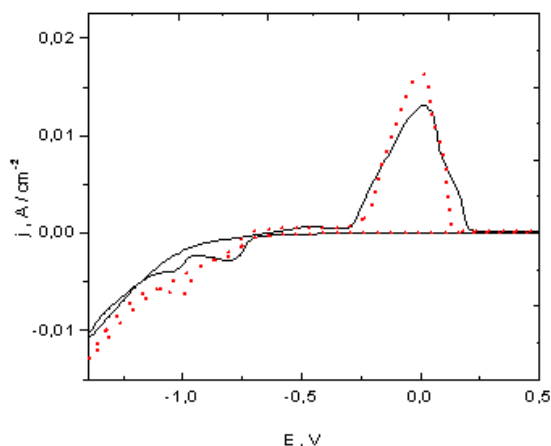
Fig. 2b. Influence of glycine on the electrode processes during electrodeposition of tin from *sulphate/gluconate electrolyte* at pH=5. C_{Sn}=5 g dm^{-3} ; $C_{Na_2SO_4}$ = 40 g dm^{-3};$C_{C_6H_{11}NaO_7}$= 50 g dm^{-3} —$C_{C_2H_5NO_2}$ = 0 g dm^{-3}; ----$C_{C_2H_5NO_2}$ = 50 g dm^{-3}; v= 20 mV s^{-1}

In the anodic processes, there is no polarization effect of the glycine ions on tin dissolution at both pH.

Cobalt deposition

The effect of the complex forming agent ($C_6H_{11}O_7Na$) on the deposition of cobalt from sulfate electrolyte is shown on Fig.3. Strong polarization of the cathodic process in the presence of $C_6H_{11}O_7Na$ is observed due to the formation of $[Co(C_6H_{11}O_7)]^+$ complex (Fig.3 dashed line). Gluconate complex of cobalt have been found to be $[Co(C_6H_{11}O_7)]^+$ in solution of pH < 7.5 with stability constant lgK= 0.57 [24]. It has been suggested that the gluconate ion is attached to Co, by coordination trough carboxyl group and one of

the adjacent hydroxyl groups [25]. The cobalt electrodeposition is accompanied with simultaneous hydrogen evolution as a side reaction. Decreased overall rate of the electrochemical reaction in presence of gluconate is registered.

In the anodic processes, the effect of the gluconate ions on cobalt dissolution is ambiguous. The anodic peak potential of the dissolution of cobalt in presence of gluconate is observed in the same potential range compared with the gluconate free electrolyte.

Glycine is a simple amino acid usually presented with the chemical formula NH_2CH_2COOH. Depending of the pH of the solution, glycine exists as a cation in acidic solution, as neutrally charged zwitterion at intermediate pH values, and as anion in basic media. It is known from basic chemistry that glycine form complexes with Co^{2+} ions in aqueous solution [26]. Formation of three complexes is possible depending on the pH of the
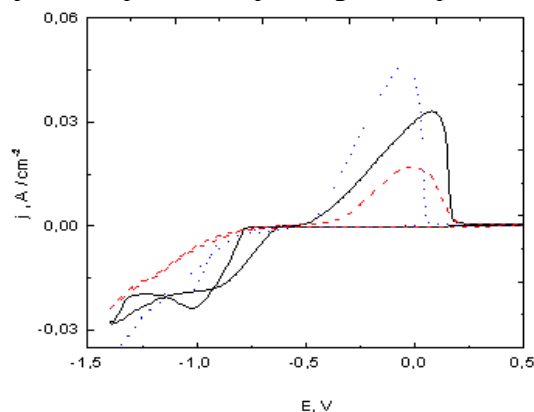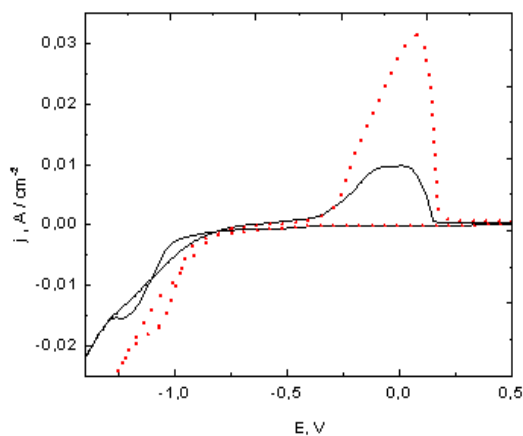

Fig. 3. Influence of glycine and gluconate on the electrode processes during electrodeposition of cobalt from *sulphate electrolyte* at pH=5. C_{Co}=5 g dm^{-3}; $C_{Na_2SO_4}$ = 40 g dm^{-3};v=20 mV s^{-1};—$C_{Na_2SO_4}$ = 40 g dm^{-3}; $^{...}C_{C_2H_5NO_2}$ =50 g dm^{-3}; ---$C_{C_6H_{11}NaO_7}$= 50 g dm^{-3}

solution$Co(NH_2CH_2COOH)^+$, $Co(NH_2CH_2COOH)_2$ and $Co(NH_2CH_2COOH)_3^-$ with stability constant lgK = 4.6, lgK = 8.4 and lgK = 10.8 respectively. The formation of Co-glycine complexes is favored at high pH in the cobalt electrolyte.

The effect of the glycine addition on the deposition of cobalt from sulfate electrolyte is shown also on Fig.3. Strong polarization of the cathodic process in the presence of NH_2CH_2COOH is observed due to the formation of $Co(NH_2CH_2COOH)_3^-$ complex (Fig.3 dot line).

In the anodic processes, the effect of the glycine ions on cobalt dissolution is registered. The anodic peak potential of the dissolution of cobalt in presence of glycine increased and is shifted to more negative values compared with the glycine free electrolyte.

Figure 4 shows the cyclic voltammetric curves obtained for cobalt electrodeposition from sulphate/gluconate electrolyte with addition of 50 g dm^{-3} glycine. The presence of glycine in this

Fig. 4. Influence of glycine on the electrode processes during electrodeposition of cobalt from *sulphate/gluconate electrolyte* at pH=5.C_{Co}=5 g dm^{-3}; $C_{Na_2SO_4}$ = 40 g dm^{-3};$C_{C_6H_{11}NaO_7}$= 50 g dm^{-3}; —$C_{C_2H_5NO_2}$ = 0 g dm^{-3}; ---$C_{C_2H_5NO_2}$ =50 g dm^{-3}; v= 20 mV s^{-1}

electrolyte has very strong depolarizing effect. Glycine acts as accelerator for cobalt reduction and does not block the active sites on the electrode surface for cobalt deposition.

The higher anodic maximum corresponds to the dissolution of increased co-amount, deposited in the cathodic period.

Tin-Cobalt alloy deposition

Figure 5 shows the cyclic voltammetric curves recorded at pH=5 in sulphate/gluconate electrolytes containing ions of both metals separately and together. During the deposition of tin two cathodic reactions are registered. During investigation of cobalt deposition from the same electrolyte in the absence of tin the occurrence of one cathodic reaction is observed. In this case, tin is the more positive component (both deposition peaks of tin, respectively the firsts peak recorded at -800 mV and the second peak recorded at -1050 mV are less cathodic (less negative) compared with the deposition peak of pure cobalt registered at -1200 mV). When cobalt is added to the solution of tin two cathodic maxima are observed. The first cathodic maximum recorded on cyclic volatmmetric curve of the alloy, which corresponds to the deposition of pure tin is polarised compared with those of pure tin and the second cathodic maximum is depolarized compared to pure cobalt. The differences between the size and the position of the peaks in the case of the alloy deposition and the deposition of the separate metals are due to the

reformation of the complexes in the alloy electrolyte, because the experiments are carried out using the same concentration of free complexing agents in all electrolytes.
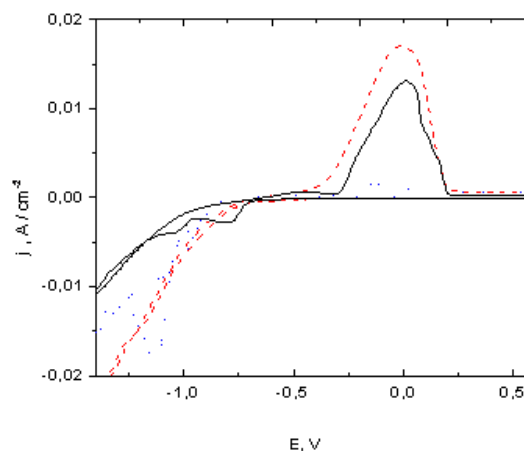
Fig. 5. CVA curves of tin, cobalt and alloy deposition from sulphate/gluconate electrolytes at pH=5 $C_{Na_2SO_4}$ = 40 g dm^{-3};$C_{C_6H_{11}NaO_7}$= 50 g dm^{-3};v= 20 mV s^{-1} —— C_{Sn} = 5 g dm^{-3}; ---- C_{Co} = 5 g dm^{-3} ;······C_{Sn} = 5 g dm^{-3}; C_{Co} =5 g dm^{-3}

Co-deposition of tin and cobalt from sulfate/gluconate electrolyte solution was mainly reported as a normal system [11]. The more positive metal, tin, was deposited preferentially.

Two anodic reactions are detected at case of dissolution of tin and one during dissolution of cobalt and the alloy. The main oxidation peak of the reaction of the alloy is in the potential range where dissolution of tin and cobalt takes place.

Figure 6 shows the cyclic voltammetric curves in sulphate/gluconate electrolytes recorded at different pH. The fresh prepared solution has pH of about 3.5. Adjusting the pH to 5 by addition of KOH results in a strong polarization effect on the first cathodic maximum during the deposition of the alloy. This maximum is connected with deposition of pure tin. It is known that the pH is the main factor that governs the rate of Sn(II) reduction. In gluconate solution the electroreduction of tin is accompanied by inhibitive adsorption that grows with pH [27]. The potential of the second cathodic reaction remains unchanged and a strong decrease of the reaction rate is registered.

Figure 7 shows the cyclic voltammetric curves at different glycine concentrations in the alloy deposition electrolyte recorded at pH=5. The increase of the glycine concentration leads to increase of reaction rate of the first cathodic reaction, and depolarization of the second cathodic reaction. During anodic scan easier dissolution of

the coating depending on the glycine concentration is observed. At pH=3.5 the same effect of glycine concentration on the deposition of the alloy is observed (not shown in the paper).
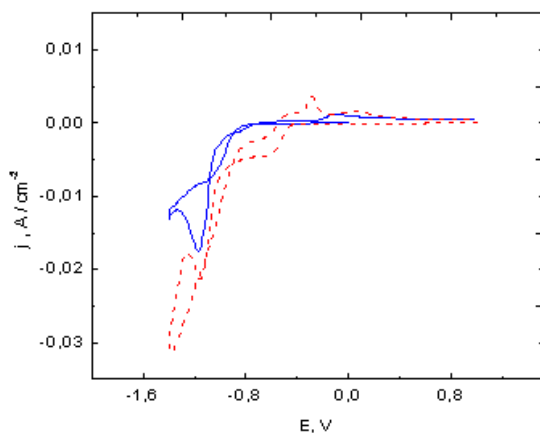

Fig. 6. Influence of the pH on the electrode processes during electrodeposition of the alloy from *sulphate/gluconate electrolytes.* $C_{Na_2SO_4}$ =40 g dm^{-3};$C_{C_6H_{11}NaO_7}$= 50 g dm^{-3}; C_{Sn} = 5 g dm^{-3}; C_{Co} =5 g dm^{-3} *v*= 20 mV s^{-1} —— pH = 5 ;---- pH = 3.5
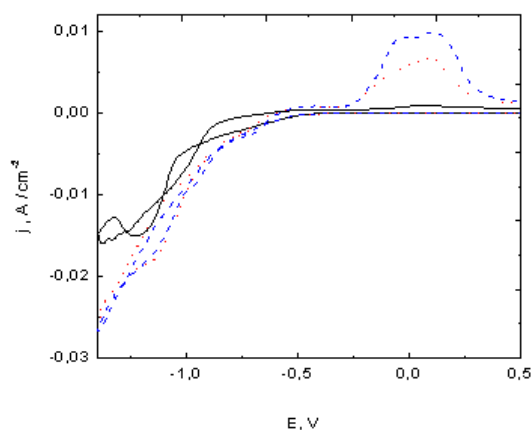
Fig. 7. Influence of glycine on the electrode processes during electrodeposition of the alloy from *sulphate/gluconate electrolyte* pH=5. $C_{Na_2SO_4}$ = 40 g dm^{-3};$C_{C_6H_{11}NaO_7}$= 50 g dm^{-3}; C_{Sn} = 5 g dm^{-3}; C_{Co} =5 g dm^{-3} *v*= 20 mV s^{-1} —— $C_{C_2H_5NO_2}$ = 0 g dm^{-3};······$C_{C_2H_5NO_2}$ = 15 g dm^{-3} ;---- $C_{C_2H_5NO_2}$ = 30 g dm^{-3}

Figure 8 shows the effect of both pH of the electrolyte and the addition of glycine in the electrolyte on the composition of the alloy. In the absence of glycine in the electrolyte at pH=3.5, pure tin coatings are deposited at low current densities. With the rise of the current density, the cobalt content in the coating increases up to about 50 wt.%. At the low pH (around 3.5) the effect of addition of glycine is more obvious, the cobalt content in the coatings rise with about 10 % compared to the cobalt content in the coating obtained from glycine free electrolyte.

Figure 9 shows SEM images of coatings deposited from glycine free electrolyte at current density 6 mA cm^{-2} and pH =3.5 (Fig.9 a)) and pH = 5.0 (Fig.9 b)) respectively. The increase of pH results in more compact morphology and globular structures formed by agglomeration of fine grains. At low pH=3.5 X-ray fluorescence analysis gives up to 40 wt. % cobalt content in the coatings. At pH=5 at the same conditions, the coatings contain 54% cobalt.
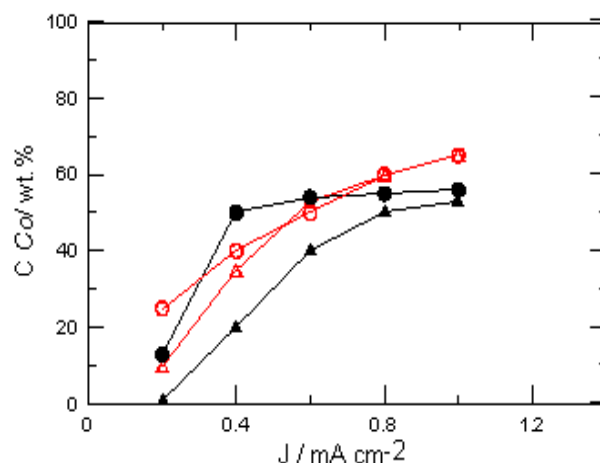
Fig. 8. Influence of the current density on the cobalt content in the alloy at different pH in presence and absence of glycine. $C_{Na_2SO_4}$ = 40 g dm^{-3};$C_{C_6H_{11}NaO_7}$= 50 g dm^{-3}; C_{Sn} = 5 g dm^{-3}; C_{Co} =5 g dm^{-3} *v*= 20 mV s^{-1} ; ● $C_{C_2H_5NO_2}$ = 0 g dm^{-3}; pH=5.0; ○$C_{C_2H_5NO_2}$ = 50 g dm^{-3}; pH=5.0; ▲$C_{C_2H_5NO_2}$ = 0 g dm^{-3}; pH=3.5; △$C_{C_2H_5NO_2}$ = 50 g dm^{-3}; pH=3.5.

Figure 10 shows SEM images of coatings deposited at current density 10 mA cm^{-2} and pH = 5 in absence (Fig.10 a)) and in presence (Fig.10 b)) of glycine. The addition of glycine in the electrolyte results in more compact morphology and very smooth surface. In absence of glycine in the electrolyte, the cobalt content in the coatings was 55%. The presence of glycine in the electrolyte results in increased cobalt content 60 wt. % cobalt.

CONCLUSIONS

Addition of glycine to the sulfate/gluconate electrolyte for electrodeposition of SnCo alloys leads to:

-Deposition of more homogeneous compact alloy coatings with better appearance.

-Glycine favors the cobalt deposition, accelerates the Co^{2+} ions reduction as confirmed by CV curves.

- Cobalt content in the alloy increases in the presence of glycine.

- Coatings with up to 65 wt. % Co can be deposited from investigated electrolyte.
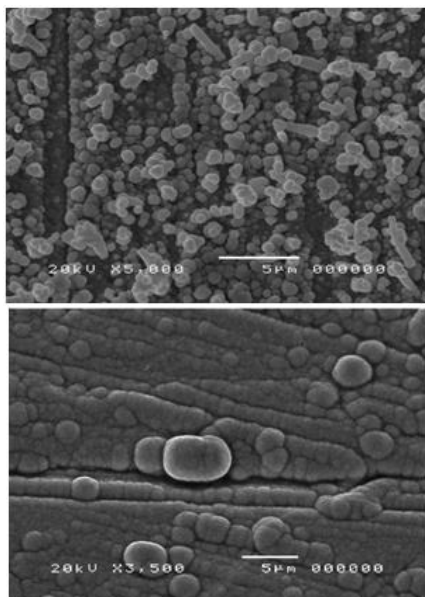
Fig. 9. Surface of tin-cobalt alloy coatings C_{Sn}= 5 g dm^{-3}; C_{Co}= 5g dm^{-3}; $C_{Na_2SO_4}$= 40 g dm^{-3}; $C_{C_6H_{11}NaO_7}$= 50 g dm^{-3} $C_{C_2H_5NO_2}$ = 0 g dm^{-3}; 6 mA cm^{-2} *(a)* pH=3.5, 40 wt. % *Co;(b)*pH=5.0, 54 wt. % *Co;*
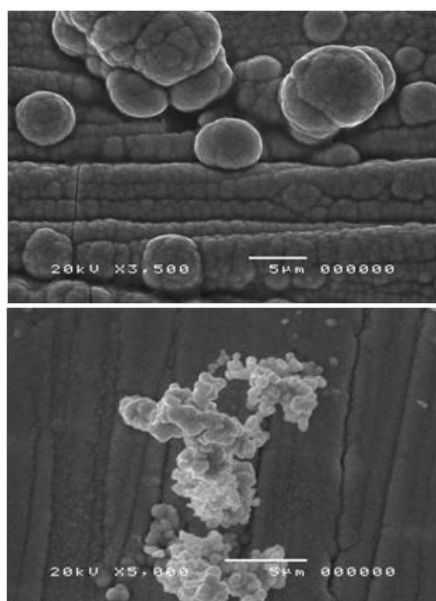
Fig. 10. Surface of tin-cobalt alloy coatings at pH=5 C_{Sn}=5 g dm^{-3}; C_{Co}=5g dm^{-3}; $C_{Na_2SO_4}$=40 g dm^{-3}; $C_{C_6H_{11}NaO_7}$=50 g dm^{-3}; 10 mA cm^{-2} *(a)*$C_{C_2H_5NO_2}$ =0 g dm^{-3}, 55wt. % *Co*; *(b)*$C_{C_2H_5NO_2}$ =50 g dm^{-3},65wt. % *Co*.

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ВЛИЯНИЕ НА ГЛИЦИН ВЪРХУ ЕЛЕКТРОХИМИЧНОТО ПОЛУЧАВАНЕ НА СПЛАВ Sn-Co ОТ ГЛЮКОНАТЕН ЕЛЕКТРОЛИТ

Т.Вълкова[*]и И.Кръстев

Институт по физикохимия, Българска академия на науките, ул. „Акад. Г. Бончев", бл. 11, 1113 София, България

(Резюме)

Изследвано е влиянието на глицин върху електродните процеси на отлагане и разтваряне на калай, кобалт и сплав калай-кобалт от глюконатно-сулфатен електролит посредством метода на циклична волтамперометрия.

Установено е, че добавянето на глицин към слабо кисел електролит за получаване на покрития от сплав калай-кобалт не оказва съществено влияние върху отлагането на калай, но има силен ефект, като при отлагането на чист кобалт, така и при отлагане на сплавта калай-кобалт. Добавянето на глицин и повишаването на концентрацията му в електролита води до нарастване на процентното съдържание на кобалт в покритията. От изследвания електролит могат да бъдат получени качествени покрития с високо съдържание на кобалт. Промяната pH на електролита и добавянето на глицин дава възможност за получаване на покрития със съдържание на кобалт от 0 до 65 тегл. %.