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Investigation of the interaction between cobalt sulfide coatings and Ag(I) ions using cyclic voltammetry in KClO$_4$ and NaOH aqueous solutions and X-ray photoelectron spectroscopy

Abstract Cobalt sulfide coatings have been investigated by means of cyclic voltammetry in 0.1 M KClO$_4$ and 0.1 M NaOH solutions and analyzed using X-ray photoelectron spectroscopy. They have been shown to contain CoS(OH), CoS and Co(OH)$_2$. After treating such Co sulfide coatings with AgNO$_3$ solution, their composition changes: both the cobalt and oxygen content decreases and Ag (up to 85 at%) appears in the coating as Ag$_2$S, Ag$_2$O and metallic Ag. Co(II) compounds react with Ag$^+$ ions according to an exchange reaction [CoS + 2Ag$^+$ + 2H$_2$O→Ag$_2$S + Co(OH)$_2$ + 2H$^+$]. In the course of the reaction of Co(OH)$_2$ with silver ions, a redox process occurs, giving metallic silver [Co(OH)$_2$ + Ag$^+$ + H$_2$O→Ag° + Co(OH)$_3$ + H$^+$ or Co(OH)$_2$ + Ag$^+$→Ag° + CoO(OH) + H$^+$]. Ag$_2$S reduction takes place at more positive potentials than Cu reduction; therefore sulfide layers of cobalt modified with silver ions, unlike unmodified ones, may be plated with Cu from both acid and alkaline electrolytes.

Keywords Cobalt sulfide coatings · Silver(I) ions · Cyclic voltammetry · X-ray photoelectron spectroscopy

Introduction

At present the coatings of non-stoichiometric copper sulfide (Cu$_{2-x}$S) are used most widely for electrochemical metallization of dielectrics and have been thoroughly investigated [1, 2, 3, 4, 5, 6, 7, 8]. However, reliable metallization of dielectrics is possible only in the event that a cycle (N) for the formation of Cu$_{2-x}$S coatings is repeated several times. Besides, direct electrodeposition

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on Cu$_{2-x}$S is possible only with those metals whose discharge potential (E) is more negative or at least equal to the Cu$_{2-x}$S reduction potential [8]. Among such metals is Ni, which can be electrodeposited on Cu$_{2-x}$S from various nickel plating electrolytes. Meanwhile, the copper electrodeposition potential is more positive than that of Cu$_{2-x}$S reduction, this being one of the causes of the difficult application of Cu$_{2-x}$S coatings in the production of printed circuits based on electrochemical deposition of copper in the holes. So, in order that the Cu$_{2-x}$S coating should be plated with copper, it must be modified [9].

At present it is known that using cobalt sulfide coatings instead of Cu$_{2-x}$S makes it possible to reduce the number of cycles for the formation of metal sulfide coatings. However, electrodeposition of copper on cobalt sulfide coating also is possible only after its modification.

After treating the cobalt sulfide coating with Ag$^+$ ions, as in the case of Cu$_{2-x}$S [9], at least a portion of its zcomponents can be substituted by ions of a more electropositive metal whose reduction would occur at potentials more positive compared with the potential of copper electrodeposition from an acid electrolyte. It is just these changes that are chosen as the object of our study.

Experimental

Deposition and modification of cobalt sulfide coatings

Electroconductive cobalt sulfide coatings were formed chemically. The substrate being coated was treated for 30 s at 20 °C with a solution of cobalt ammoniate (0.16 M CoSO$_4$·7H$_2$O + 0.12 M (NH$_2$OH)$_2$·H$_2$SO$_4$ + 25% NH$_3$, pH≈11). When rinsing with water, the Co(II) compounds adsorbed on the surface being coated are hydrolyzed to insoluble compounds, which cohere well with the substrate. In the second stage the surface being coated is treated for 30 s at 20 °C with 0.13 M sodium sulfide solution and rinsed with water [10]. This is one cycle of sulfidation. In order to obtain a thicker coating, one needs to increase the number of cycles. The formed Co sulfide coatings were modified by keeping them during 30 or 120 s at 20 or 40 °C in freshly prepared AgNO$_3$ (1.7 and 30 mM) solution.

The coatings were formed on a CY-1200 glassy carbon electrode. The working area of the electrode (1 cm²) was prepared as follows: before forming the Co sulfide coating the plate of glassy carbon (50×15×2 mm) was covered by poly(vinyl chloride) lacquer (PVCh-10), leaving an area of 10×10 mm unlacquered, and then the coating was formed on the entire plate. After that the sulfidized lacquer was removed mechanically, and the same area was insulated again with the same lacquer. Only the area of 1 cm² remained covered with Co sulfide and electrochemically active.

Electrochemical measurements

Electrochemical behaviour of the coatings was studied by cyclic voltammetry in 0.1 M $KClO_4$ and 0.1 M NaOH solutions at a potential sweep rate of 0.05 V s^{-1}. A platinum screen served as an auxiliary electrode and an Ag|AgCl|KCl (sat) electrode as the reference electrode. All the potentials (E) are quoted with respect to the SHE. The measurements were performed using a potentiostat PS-305 (Elchema, USA), using the data acquisition software Voltscan.

X-ray photoelectron spectroscopy measurements

Co sulfide coatings, both untreated and treated with Ag(I) ions, were studied using X-ray photoelectron spectroscopy (XPS). The XPS spectra were recorded by a spectrometer (Escalab Mk II, VG Scientific, UK) using Mg anode radiation (1253.6 eV, the characteristic radiation energy of Mg K$_\alpha$), the capacity of which was 300 W. A vacuum of 1.33×10^{-6} Pa was kept in the analysis chamber. Distribution of the elements depending on the depth was determined by using an Ar$^+$ ion gun with an ion stream energy of ~1.0 keV. The samples in the preparation chamber were etched in a vacuum of 6×10^{-3} Pa and a current of ~20 µA/cm². This corresponds to an etching rate of ~5 nm/min. The maximum accuracy of the method is ±0.1 at%. When investigating chemical cobalt sulfide coatings by XPS, photoelectron spectra of the elements (Co 2p$_{3/2}$, O 1s, S 2p, Ag 3d$_{5/2}$) were recorded. Empirical sensitivity factors of these elements were taken from the literature [11] and the spectra obtained were compared with standard ones [12]. In the paper the data of the solid cobalt sulfide coating are presented. Reproducibility of the distribution of the elements depending on the depth was found to be very good. More obvious scattering of the elements was observed only at the surface of the coatings.

Results and discussion

Cyclic voltammograms (CVs) of cobalt sulfide coatings on glassy carbon recorded in 0.1 M NaOH solution are presented in Fig. 1. The corresponding electrochemical reactions have been described [13]: in the potential region of peak K_1, Co(III) [CoS(OH)] is reduced to Co(II) compounds [CoS and Co(OH)$_2$] that are reduced to metallic cobalt in the potential region of hydrogen evolution; in the region of peak A_1 the metallic cobalt is oxidized to Co(II) compounds and in the region of peak A_2 to Co(III) compounds.

After treating a cobalt sulfide coating with AgNO$_3$ solution at 20 °C, a change in the CV is observed as soon as 30 s (Fig. 2). The greater the duration of the treatment, the more obvious is the change. Peak K_1, which is attributed to Co(III) reduction to Co(II) [13], disappears and instead of it peak K_{1m} appears. In 0.1 M NaOH solution (Fig. 2), peak K_{1m} is shifted with respect to peak K_1 to the cathodic side by 100–120 mV. After
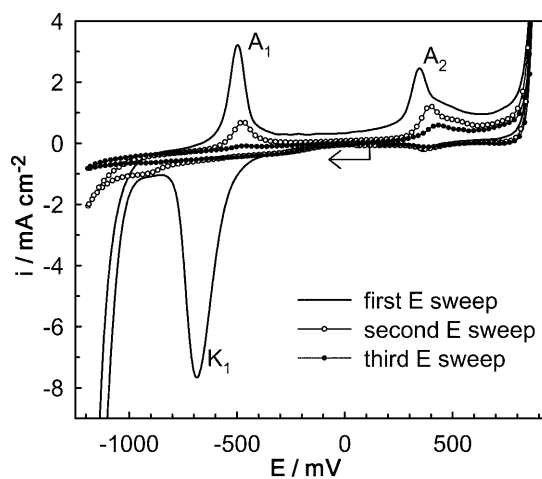

Fig. 1 CVs of Co sulfide coatings formed on a GC electrode by two cycles recorded in 0.1 M NaOH solution. In Figs. 1, 2, 3, 4, 5 the potential sweep rate $v = 50$ mV/s, $T = 20$ °C
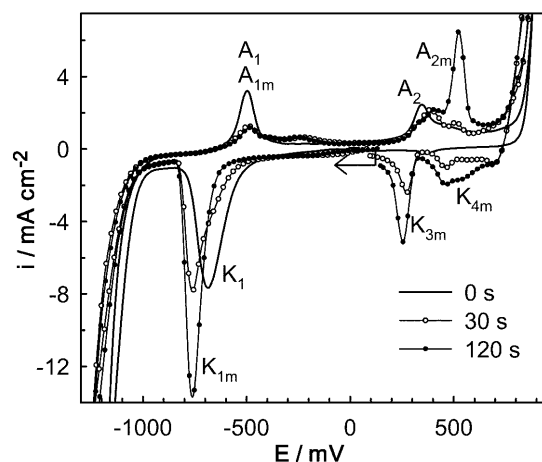

Fig. 2 CVs of Co sulfide coatings formed on a GC electrode by two cycles as a function of duration of treatment in 30 mM AgNO$_3$ solution; recorded in 0.1 M NaOH solution

treatment, peak A_2, attributed to Co(II) oxidation to Co (III) [13], was found to become lower in the anodic CV portion. Instead, peak A_{2m}, representing the oxidation of metallic silver to Ag$_2$O, appears (Table 1) [9]. When returning to the cathodic side, two new current peaks, K_{3m} and K_{4m}, appear and grow with increasing duration of the treatment (Fig. 2).

These changes, when compared with the unmodified coating, are less pronounced when CVs of Co sulfide coatings treated with Ag$^+$ ions at 20 °C are recorded in neutral (0.1 M KClO$_4$) medium (Fig. 3). However, on increasing the temperature of the AgNO$_3$ solution (from 20 to 40 °C), peak K_{1m} was observed not only to increase but also to shift by ~100 mV to the cathodic side with respect to K_1. The observed increase of peak K_{1m}, compared with peak K_1, with increasing temperature (Fig. 3) or with increasing duration of the treatment (Fig. 2), can be explained by the fact that peak K_1

Table 1 Redox processes of Co sulfide coating formed on GC and treated with 30 mM AgNO$_3$ solution, occurring in alkaline (0.1 M NaOH) medium. Duration of treatment: 120 s; $T = 30$ °C

Peak	Process	Potential of peak (V)	$E°$ (V)	Ref
K$_{1m}$	Ag$_2$S + 2e$^-$→2Ag° + S^{2-}	−0.75 to −0.80	−0.66	[9, 14, 15, 16, 17, 18, 19, 20, 21, 22]
K$_{3m}$	Ag$_2$O + H$_2$O + 2e$^-$→2Ag° + 2OH$^-$	0.3–0.32	0.345	[9, 14]
K$_{4m}$	Ag$^+$ + e$^-$→Ag°	0.67–0.70	0.799	[9, 14]
A$_{1m}$	2Ag° + S^{2-}→Ag$_2$S + 2e$^-$	−0.64 to −0.65	−0.66	[9, 14]
A$_{2m}$	2Ag° + 2OH$^-$→Ag$_2$O + H$_2$O + 2e$^-$	0.45–0.46	0.345	[9, 14]

represents only the reduction of Co(III) present in the coating to Co(II) [13]. Meanwhile, the current maximum representing the reduction of CoS present in the coating is masked by the hydrogen evolution current. It is just the CoS interaction with Ag(I) that can lead to an increase in the K$_{1m}$ current maximum.

In the anodic region the changes in the CVs are more pronounced: at potentials of 880–900 mV, instead of current peak A$_2$ one can observe a new double current peak A$_{2m}$, which is shifted with respect to the initial A$_2$ to the anodic direction by 130–40 mV ($E = 1130$–1150 mV) (Figs. 2 and 3). The changes in the CVs undoubtedly show that the initial Co sulfide coating is modified by Ag$^+$ ions.

In the potential region of current peak K$_{1m}$ the reduction of the formed Ag$_2$S to Ag° is unquestionable (Table 1):

$$Ag_2S + 2e \rightarrow 2Ag° + S^{2-} \tag{1}$$

Current peak A$_{1m}$, appearing in the anodic region, is related to the interaction of metallic Ag with S^{2-} ions that had been formed in the potential region of current peak K$_{1m}$ and failed to be removed from the surface. Peak A$_{1m}$ coincides with peak A$_1$, which represents the oxidation of Co, formed in the course of CoS reduction in the potential region of hydrogen evolution, to Co(II) [Co(OH)$_2$] both in alkaline (NaOH) and neutral (KClO$_4$) media (Figs. 2 and 3). This peak decreases if,

during recording of the cathodic portion of the CV, the potential is kept constant for ∼1 min in the hydrogen evolution potential region ($E < -1100$ mV). In this case one can observe an increase in peak A$_{2m}$, attributed to oxidation of metallic silver to Ag$_2$O (Table 1). When returning to the cathodic region, in alkaline solution two peaks appear: the unclear current peak K$_{4m}$ and the narrow, clearly pronounced peak K$_{3m}$, the first being related to the reduction of Ag$^+$ to Ag°, and the second one to the reduction of Ag$_2$O, formed in the A$_{2m}$ potential region, to Ag° (Fig. 2, Table 1) [9].

In the course of the second potential sweep, neither current peak K$_1$ nor current peak K$_{1m}$ appear (Fig. 4). This shows that, in the potential region of K$_1$ and K$_{1m}$, compounds of both cobalt and silver, whose reduction occurs in the potential region of these peaks, are reduced completely (Table 1). It is interesting that an unclear peak K$_1$ is observed after treating the Co sulfide coating with Ag$^+$ ions for 30 s (Fig. 4), which is undoubted testimony to incomplete exchange between Co(III) and Ag(I) compounds. After treating the coating with Ag$^+$ for 120 s the exchange is completed and current peak K$_{1m}$ becomes narrower (Fig. 5) compared with incomplete substitution of Co(III) (peaks K$_1$ and K$_{1m}$, Fig. 4). However, the fact that even in this case an unclear peak A$_2$ remains (compare A$_2$ in Figs. 4 and 5) is indicative of Co(III) compounds left in the coating.
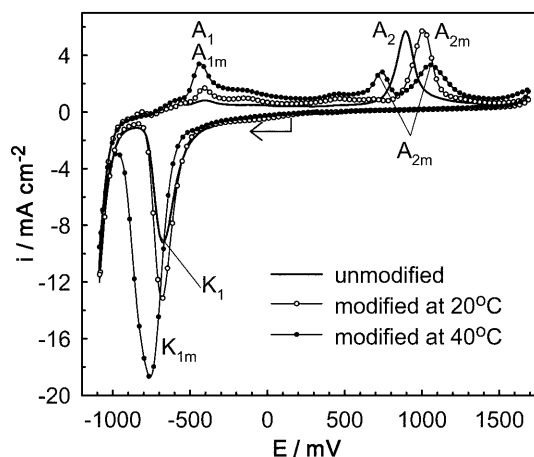

Fig. 3 CVs of Co sulfide coatings formed on a GC electrode by two cycles as a function of temperature in 30 mM AgNO$_3$ solution. Duration of treatment 120 s; recorded in 0.1 M KClO$_4$ solution

Fig. 4 CVs of Co sulfide coatings formed on a GC electrode by two cycles and kept for 30 s in 30 mM AgNO$_3$ solution; recorded in 0.1 M NaOH solution
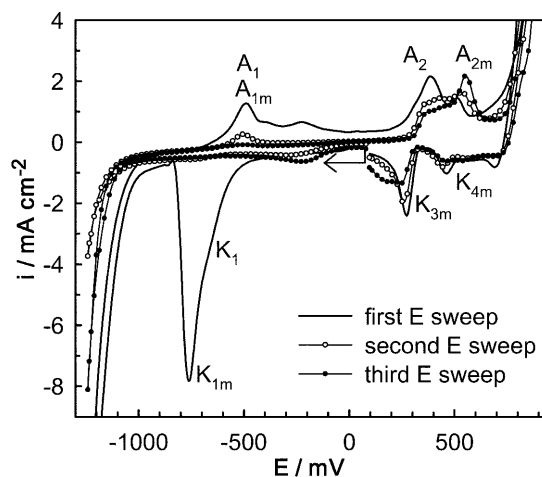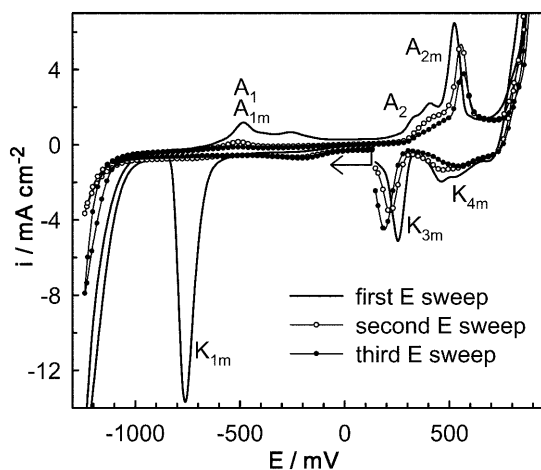
Fig. 5 CVs of Co sulfide coatings formed on a GC electrode by two cycles and kept for 120 s in 30 mM AgNO₃ solution; recorded in 0.1 M NaOH solution

When investigating the composition of Co sulfide coatings by XPS, attention was paid to the distribution of Co, S and O both on the surface and in the depth of the coating. As is seen from Table 2, in both the unmodified and the modified coating, especially on the surface, a large amount of oxygen (up to ~43 at%) is found.

On the surface, $CoS(OH)$ was formed, while in the case of coating etching, in a depth up to ~2 nm, $Co(OH)_2$ and $CoSO_3$ are believed to be formed as a consequence of the redox reaction:

$$6CoO(OH) + 6S^{2-} + 3H_2O \rightarrow CoSO_3 + 5CoS + 12OH^- \quad (2)$$
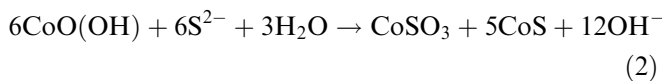
As is seen from the stoichiometry of this reaction, cobalt in the form of $CoSO_3$ in the sulfide coating should not exceed 1/6 of all the cobalt present in the coating. Therefore one can suppose that only a very small percentage of all the cobalt present in the coating is in the form of $CoSO_3$. Most probably it is formed only on the surface of the coating and is reduced in the potential region of peak K_1.

The compound $CoS(OH)$ can be formed in two ways: (1) in the course of oxidation of Co(II) sulfide to Co(III) and the following hydrolysis to $CoS(OH)$; (2) in the course of direct interaction between S^{2-} ions and $CoO(OH)$ [25]:

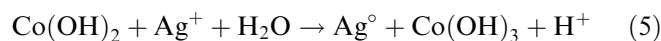$$CoO(OH) + S^{2-} + H_2O \rightarrow CoS(OH) + 2OH^- \quad (3)$$
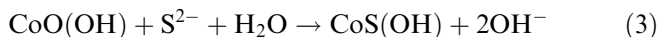
After treating such a coating of Co sulfide with AgNO₃ solution, a complete change in its composition is observed: the amount of cobalt and oxygen considerably decreases and Ag_2S, Ag_2O and metallic Ag develop in the coating (Table 2). The measured E_B for Ag $3d_{5/2}$, both on the surface and in the studied depths, was 368.0 ± 0.1 eV. This supports the conclusion made from Figs. 1, 2, 3, 4, 5 that the components of the coating are
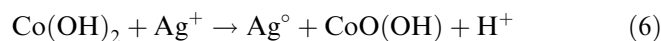
substituted by Ag^+ ions. Bearing in mind the fact that the solubility product of Ag_2S is very low ($L_{Ag_2} = 5.7 \times 10^{-51}$) compared with those of cobalt sulfides ($L_{CoS} = 3.1 \times 10^{-23} - 1.9 \times 10^{-27}$), it can be believed that an exchange reaction takes place between Co(II) compounds and Ag^+ ions, forming Ag_2S:

$$CoS + 2Ag^+ + 2H_2O \rightarrow Ag_2S + Co(OH)_2 + 2H^+ \quad (4)$$
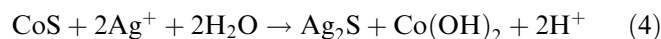
However, the fact that metallic silver is found in the coating shows that not only the exchange reaction 4 occurs in the presence of Co(II), but the oxidation-reduction process which forms Ag° and $Co(OH)_3$ ($E^o_{Co(OH)_3/Co(OH)_2} = 0.17$ V, $E^o_{Ag^+/Ag} = 0.799$ V) occurs in the coating as well:

$$Co(OH)_2 + Ag^+ + H_2O \rightarrow Ag^\circ + Co(OH)_3 + H^+ \quad (5)$$

or:

$$Co(OH)_2 + Ag^+ \rightarrow Ag^\circ + CoO(OH) + H^+ \quad (6)$$

as the data of the XPS studies show that $CoO(OH)$ is found to be present on the coating surface after treatment with Ag^+ ions.

Subsequently, reducing the modified coating in 0.05 M H_2SO_4 solution (E was swept from its stationary value to the hydrogen evolution E region and was kept constant in this region for 2 min) the water-insoluble Co(II) and Co(III) hydrolysis products dissolve and are removed from the coating into the bulk of the solution (Table 2). If cobalt sulfide compounds remained in the coating, then after their reduction the presence of metallic Co would be recorded by XPS. However, according to the data of Table 2, after the reduction only a trace of cobalt is detected on the coating surface (~0.3 at%). This indicates that all the cobalt present in the sulfide compounds was substituted by Ag^+ ions under the conditions of our experiments.

From the data of Table 2 and the spectra obtained, we were unable to calculate how much metallic silver is in the coating. A large amount of oxygen, which always accompanies cobalt compounds, is a serious handicap to such a study. However, from Eqs. 5 and 6 it could be inferred that, after the oxidation-reduction reaction, all the silver must be in the metallic state. The main part of the cobalt present in the coating is combined in Co(III) compounds. Therefore, Ag should not exceed a few percent. Moreover, a considerable part of it reacts with oxygen in the air to produce Ag_2O (Table 2):

$$2Ag^\circ + 1/2O_2 \rightarrow Ag_2O \quad (7)$$

After the modification stage a new coating appears, which is a silver sulfide coating. All the silver compounds present in the coating are reduced cathodically and, only after that, copper discharges cathodically from the acidic copper plating electrolyte on the metallic surface.

Table 2 Data from X-ray photoelectron spectroscopy for Co sulfide coating before and after treatment with Ag$^+$ ions

Etching	Element	Content (at%)	Binding energy (eV)	Most probable composition of the coating	Co/S	Co/(S+O)
Co sulfide coating, $N = 3$, $T = 20$ °C						
Surface	Co	16.2	778.7, 780.1, 780.6	CoS, CoSO$_3$, CoS(OH),	0.40	0.20
	O	43.2	531.0, 533.3	Co(OH)$_2$		
	S	40.6	162.5, 167.9			
30 s, 20 µA	Co	33.3	778.1, 780.4	CoS, CoSO$_3$, Co(OH)$_2$	0.74	0.50
(~1.0 nm)	O	21.7	531.0, 529.7			
	S	45.0	162.3, 167.0			
60 s, 20 µA	Co	41.1	778.0, 780.4	CoS, CoSO$_3$, Co(OH)$_2$	1.00	0.70
(~2.0 nm)	O	18.2	531.0, 529.7			
	S	40.7	162.3			
120 s, 20 µA	Co	47.2	778.1, 780.4	CoS, CoS(OH)	1.74	0.89
(~4.0 nm)	O	25.6	531.0, 529.7			
	S	27.2	162.3			
240 s, 20 µA	Co	43.8	778.1	CoS, CoS(OH)	2.11	0.78
(~8.0 nm)	O	35.4	531.0, 529.7			
	S	20.8	162.3			
Co sulfide coating kept for 120 s in 30 mM AgNO$_3$ solution, $T = 40$ °C						
Surface	Co	4.1	778.9	CoS, CoO(OH), Co(OH)$_3$,	1.53	1.09
	O	13.6	531.1, 532.6	Ag$_2$O, Ag$_2$S, Ag^0		
	S	34.2	161.7, 167.7			
	Ag	48.1	367.9			
30 s, 20 µA	Co	7.5	778.3, 780.6	CoS, Co(OH)$_2$, Ag$_2$O, Ag$_2$S, Ag^0	1.99	1.79
(~1.0 nm)	O	4.6	531.1			
	S	31.2	161.5			
	Ag	56.7	367.9			
60 s, 20 µA	Co	11.3	778.7, 780.4	CoS, Co(OH)$_2$, Ag$_2$O, Ag$_2$S, Ag^0	2.20	1.91
(~2.0 nm)	O	5.8	530.2			
	S	28.7	161.5			
	Ag	54.2	368.0			
120 s, 20 µA	Co	14.1	778.3, 780.6	CoS, Co(OH)$_2$, Co(OH)$_3$, Ag$_2$O,	2.40	1.98
(~4.0 nm)	O	6.2	529.7, 531.1	Ag$_2$S, Ag^0		
	S	27.4	161.7			
	Ag	52.3	368.1			
Co sulfide coating kept for 120 s in 30 mM AgNO$_3$ solution ($T = 40$ °C) and reduced in 0.05 M H$_2$SO$_4$ solution						
Surface	Co	0.3	778.3	CoS, Ag$_2$O, Ag$_2$S, S, Ag^0	0.03	0.06
	O	35.0	532.0			
	S	9.7	161.5, 164.5			
	Ag	55.0	368.1			
30 s, 20 µA	Co	0.0	–	–, Ag$_2$O, Ag$_2$S, Ag^0	–	–
(~1.0 nm)	O	8.5	530.6, 532.2			
	S	9.1	161.8			
	Ag	82.4	368.1			
120 s, 20 µA	Co	0.0	–	–, Ag$_2$O, Ag$_2$S, Ag^0	–	–
(~4.0 nm)	O	5.5	532.5			
	S	9.0	161.7			
	Ag	85.5	238.1			

Conclusion

By CV (in 0.1 M KClO$_4$ and 0.1 M NaOH solutions) and XPS, cobalt sulfide coatings have been shown to contain CoS, CoS(OH), Co(OH)$_2$. After treating such Co sulfide coatings with AgNO$_3$ solution, their composition changes considerably: the amount of cobalt and oxygen substantially decreases and silver (up to 85 at%) appears as Ag$_2$S, Ag$_2$O and metallic Ag.

Co(II) compounds react with Ag$^+$ ions to produce Co(OH)$_2$. Subsequently, Co(OH)$_2$ reacts with silver ions in a redox process, giving metallic silver. Ag$_2$S reduction takes place at more positive potentials than Cu reduction; therefore, sulfide layers of cobalt modified with silver ions, unlike unmodified ones, may be plated with Cu from both acid and alkaline electrolytes.

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