

Metal recovery of solid metallurgical wastes. Galvanostatic electroextraction of copper from sulphate electrolytes containing Zn^{2+} and Fe^{2+} ions

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The electroextraction of copper from acidic (130 g/L H_2SO_4) sulphate electrolytes in the presence of zinc or ferrous ions was studied by means of galvanostatic methods. The coatings, deposited from electrolytes containing 1 g/L Cu^{2+} and 50 g/L Zn^{2+} , are composed predominantly of Zn (80-90%). The coatings are black and powdery. Pure Cu coatings are produced at 1 A/dm² in electrolytes containing 10 g/L Cu^{2+} and 50 g/L Zn^{2+} . The deposits are light red, smooth and semi-bright. Current efficiency of Cu deposition at 2 A/dm² from electrolytes containing 10 g/L Cu^{2+} abruptly declines with the increase in Fe^{2+} concentration. It is higher than 90% and is practically independent of Fe^{2+} concentration during deposition from electrolytes containing 50 g/L Cu^{2+} . The coatings obtained from electrolytes containing 10 g/L Cu^{2+} in the presence of Fe^{2+} ions are dark red, rough and math and those obtained from electrolytes containing 50 g/L Cu^{2+} are light red, smooth and bright. The results of the microprobe analysis indicate that in both cases the coatings are of pure copper.

Keywords: copper; zinc; iron; electrodeposition; electroextraction

INTRODUCTION

After a number of pyro- or hydrometallurgical ore treatments, large amounts of wastes with high metal content remain (Table 1).

There are numerous papers dedicated to the recovery of metals by electrolysis from scrap metal pickling wastewater [10], gold plant waste streams [11], spent copper-cyanide electroplating electrolytes [12], and stripping solutions in the manufacture of printed circuit boards [13]. Copper electrodeposition onto porous, fluidized and spouted bed electrodes and the treatment of copper-containing waste streams is studied by J. W. Evans *et al.* [14]. A study of Fe removal from Cu leachate by limestone precipitation followed by direct electrowinning of Cu has been conducted by B. Zhang [15]. The recovery of Cu from printed circuit board scraps, containing (in wt. %): Cu (24.3-30.2), Fe (0.08-0.18) and Sn (2.5 - 4.9) by mechanical processing and electrometallurgy is demonstrated by H. M. Veit *et al.* [16]. The recovery of Cu and Sn from waste stripping solutions containing 2–40 g/L Cu and 150 g/L SnO_2 is studied by S. Roy and R. Buckle [17]. M. Aghazadeh *et al.* [18] developed the fundamentals of a method for the direct recovery of Cu from brass (Cu–30 wt.% Zn) scrap based on simultaneous electrolytic dissolution of

the scrap at the anode and electrodeposition of Cu at the cathode in an acidified sulfate electrolyte. L. Petkov and B. Spasov [19] found out that electrolytic Cu extraction from dilute H_2SO_4 solutions on a pulsating cathode permits complete Cu extraction from these solutions. L. Petkov and I. Dardanova [20] established that electrolysis from dilute H_2SO_4 solutions by a fluidized bed cathode reduces the Cu content from 1 g/L to 0.2-0.5 mg/L.

These methods, however, are inapplicable to the extraction of metals from solid wastes like powders, cakes, dusts, drosses, scraps, etc., where the content of non-ferrous metals is much higher. Though these hard waste products contain Cu and Zn in commensurate amounts, there are no data available in the literature reporting of attempts to electroextract them separately.

In the literature we have met only one paper concerning the influence of little amounts of Zn^{2+} on Cu electrodeposition. Muresan *et al.* [1] have observed that during electrowinning of Cu from wastes the deposits morphology slightly differs in the presence of Zn^{2+} . This suggests that Zn^{2+} ions are electrosorbed at the interface and slightly modify the effective surface of the copper deposits. The changes occur at small Zn^{2+} additions and there are no significant differences between the deposits obtained with Zn^{2+} concentrations of 0.05 and 0.5 g/L.

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Table 1.

Waste	Metal content, wt.%
“Blue powder” that results from condensing furnace gases during the thermometallurgical processing of non-ferrous ores.	Zn (25-41), Pb (20-25), Fe (3-5), Cu (0.5-1) [1, 2].
Cakes obtained during purification of the electrolytes for zinc electrowinning:	
1. Copper cake	Cu (36-54), Zn (5-10) [3].
2. Copper-cadmium cake	Cu (10), Zn (30), Cd (12) [3].
3. Collective cake	Cu (5.8), Zn (35.9), Cd (7.2) [3].
4. Cobalt-nickel cake	Cu (25), Zn (20), Cd (3) [3].
5. Lead-zinc cake	Zn (17-22), Pb (4.5-7), Cu (1.8-4.5), Fe (23-27) [3].
Flue dusts at a secondary copper smelter treated in the electrowinning zinc plant.	Zn (40-65), Cu (1-6), (Pb 6-20), Fe (0.1-1.5) [4].
Flue zinc dust in smelting processes.	Zn (19.4), Fe (24.6), Cu (0.42) [5].
Mixture of zinc scrap and zinc dross resulting from the zinc cathode industry.	Zn (51.01), Cu (2.7), Fe (1.6), Pb (4.28) [6].
Cake from a Waelz kiln processing zinc-lead carbonate ores.	Zn (11.3), Fe (8.3), Pb (24.6) [7].
Beta cake at the cadmium plant in the cobalt removing stage.	Zn (8.0-9.5), Co (0.5-1.4), Fe (2.3 – 5.6), Cu (1.1) [8].
Cementates obtained during the treatment of the Zn sulphate leach liquor with As ₂ O ₃ and Zn powder.	Cu (28.6), Zn (22.4) [9].

We found only three papers that discuss the influence of ferric and ferrous ions on the electroextraction of Cu from electrolytes with low metal concentration. Dew and Phillips [21] studied their effect on the cathodic and anodic reactions for dilute (5 g/L) acid CuSO₄ electrolytes with varying concentrations and mixtures of reacting species. The cathodic reduction of Fe³⁺ to Fe²⁺ leads to a significant decrease in current efficiency of deposition at high Fe³⁺ concentrations. Re-oxidation of Fe²⁺ to Fe³⁺ at the anode will compound the effect of Fe³⁺ concentration on the current efficiency. The same authors [22] also investigated the effect of Fe²⁺ and Fe³⁺ on the efficiency of Cu electrowinning from solutions containing less than 2 g dm⁻³ Cu²⁺, with an equivalent or higher concentration of iron as Fe²⁺ and Fe³⁺. Experiments showed that the current efficiency decreased in proportion to the increase in Fe³⁺ concentration. The work has shown that the Chemelec cell can achieve reasonable efficiencies for direct electrowinning from dilute leach liquors. Das and Gopala Krishna [23] studied the influence of Fe³⁺ concentration (varied from 0.5 to 6.0 g/L) on current efficiency, power consumption and cathode quality during Cu electrowinning in an open channel cell. A decrease in current efficiency started with increasing Fe³⁺ concentration at each of the flow rates studied. When Fe²⁺ was added to a Cu²⁺ - Fe³⁺ electrolyte, the harmful effect of Fe³⁺ on the current efficiency was reduced. More than 90% current efficiency

may be achieved if the Fe³⁺/ Fe²⁺ ratio is maintained ≤ 1. A marginal increase in the current efficiency was observed at an increase in Cu²⁺ concentration. This may be due to the fact that upon increasing Cu²⁺ concentration in the bath, the solution viscosity increases, which impedes the distribution of Fe³⁺ over the cathode surface. A decrease in current efficiency was observed when Fe³⁺ concentration was increased from 1.0 to 2.0 g/L at each H₂SO₄ concentration.

The literature review shows that the problem concerning the recovery of metals by electrolysis from hard wastes is neglected.

The aim of this paper is to study the influence of electrolyte composition and electrolysis conditions on the process of metal recovery from acidic sulphate electrolytes containing Cu²⁺, Zn²⁺ and Fe²⁺ ions in ratios close to the proportion of these metals in the solid metallurgical wastes.

EXPERIMENTAL

Galvanostatic deposition was carried out in a 0.5 dm³ bath on rolled copper cathodes (5.0 cm²) at current densities of 0.5, 1, 2 or 5 A/dm² using two Pb-Ag (1%) anodes. Cu cathodes were degreased in an ultrasonic bath and etched in dilute (1:1) solution of HNO₃. The potentials of deposition were measured using mercury/mercurous sulphate electrode in 0.5M H₂SO₄ (SSE), its potential vs. NHE being +0.720 V.

Table 2. Cathodic potentials, Cu and Zn content (in wt.%), phase composition and surface appearance of coatings obtained after 30 min deposition on copper cathodes.

Electrolyte composition, g/L	i, A/dm ²	E, V Cu cath.	Deposit composition, %		Coating appearance
			Cu	Zn	
Cu ²⁺ -1	1	-1.425	16.2	83.8	black powdery
Zn ²⁺ -50	5	-1.665	12.0	88.0	black powdery
H ₂ SO ₄ -130					
Cu ²⁺ -10	1	-0.460	100.0	0.0	orange smooth semi-bright
Zn ²⁺ -50	5	-0.655	95.1	4.9	dark red spongy brittle
H ₂ SO ₄ -130					

The applied current densities in this study are adequate to the conditions for industrial metals plating. Usually plant Cu electroextraction or electrorefining is carried out at current densities of 1 - 3 A/dm², whereas the current density (5 A/dm²) is a plant condition for Zn electroextraction. Cu²⁺, Zn²⁺ and Fe²⁺ concentrations were selected taking into account the ratio between these metals in waste products generated from nonferrous metallurgical industry.

Cu²⁺ ions (as CuSO₄·5H₂O) were added to electrolytes containing 220 g/L ZnSO₄·7H₂O (50 g/L Zn²⁺) and 130 g/L H₂SO₄. Fe²⁺ ions (as FeSO₄·7H₂O) were added to electrolytes containing 10 or 50 g/L Cu²⁺ and 130 g/L H₂SO₄. Both Cu²⁺ and Fe²⁺ ions were added to electrolytes containing 220 g/L ZnSO₄·7H₂O (50 g/L Zn²⁺) and 130 g/L H₂SO₄.

Metal deposition was carried out at a practically constant concentration of Cu²⁺, Zn²⁺ or Fe²⁺ ions. The Cu²⁺ concentration decreases only by 0.05 – 0.4% after 30 minutes and by 0.5 - 4% after 5 hours of deposition.

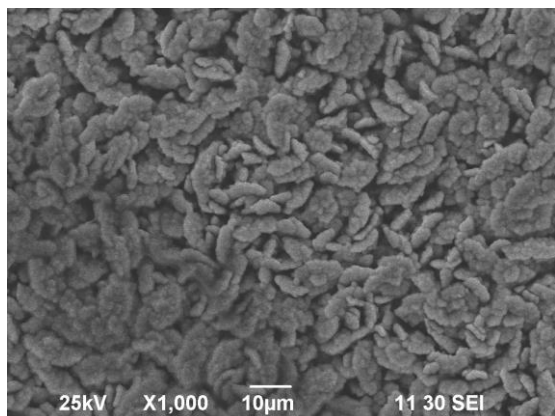
X-ray powder diffraction patterns for phase identification (characterization) of the deposits were recorded in the angle interval 20 - 110 (2θ) on a Philips PW 1050 diffractometer equipped with Cu Kα tube and scintillation detector. The surface morphology of the deposits was examined and EDX Analysis was performed by scanning electron microscopy (SEM) using a JEOL JSM 6390 microscope.

Electroextraction of Cu in the presence of Zn²⁺ ions.

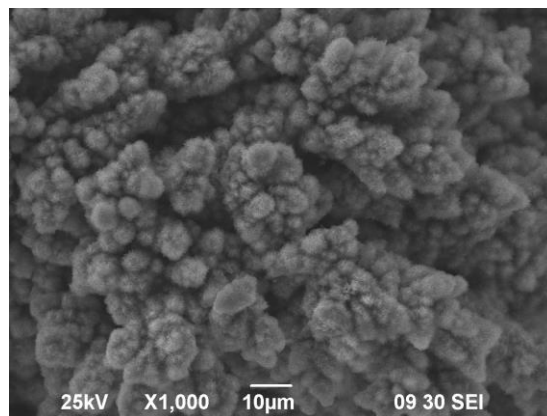
The data in Table 2 show that an increase in Cu²⁺ concentration in the electrolyte results in a decrease in the Zn content in the deposits. On the contrary, an increase in the current density yields an

increase in the content of Zn in the coatings. In electrolytes with low concentration of Cu²⁺ (1 g/L), the cathodic potentials are highly negative at both current densities, which facilitates the deposition of Zn. Besides, at the low Cu²⁺ concentration level, deposition of Cu occurs at a limiting diffusion current density (about 2 mA/cm²), which probably further decreases on longer deposition. This finding could explain the low content of Cu in the obtained coatings. There are no such diffusion limitations for the Zn²⁺ ions and hence this metal is deposited at the current density typical of electrochemical kinetics. At the higher Cu²⁺ concentration (10 g/L) the process of Cu deposition is not impeded by diffusion limitations and becomes predominating, as Cu is a more electropositive metal. At 1 A/dm², when the cathodic potential is between -0.460 and -0.485 V, a pure Cu coating is deposited, whereas at 5 A/dm², when the potential is more negative, the obtained coatings contain 4.9% Zn phase as well. Figures 1a and b show SEM pictures of coatings obtained after 30 min deposition at 1 (1a) or 5 (1b) A/dm² in electrolytes containing 1 g/L Cu²⁺, 50 g/L Zn²⁺ and 130 g/L H₂SO₄. In both cases the deposits are composed predominantly of Zn (between 80 and 90%). The coatings are black in color and powdery. Figures 2a and 2b show SEM pictures of coatings obtained after 30 min deposition from electrolytes containing 10 g/L Cu²⁺, 50 g/L Zn²⁺ and 130 g/L H₂SO₄. The coatings obtained at 1 A/dm² (2a) are light red in color with smooth and semi-bright surface. They are composed of pure Cu. The coatings deposited at 5 A/dm² (2b), however, are dark red, spongy and brittle. The coatings composition is 95.1% Cu and only 4.9% Zn.

Figures 3a and b show the SEM micrographs of pure copper coatings obtained after 30 min



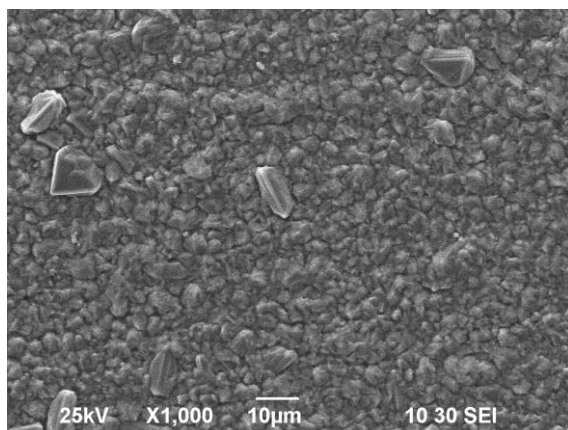
(a)



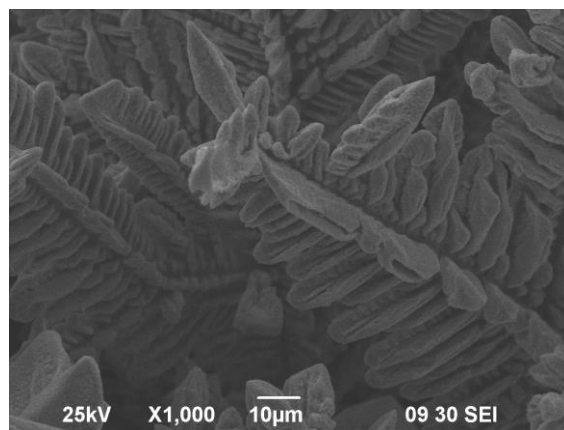
(b)

Fig. 1a. SEM micrographs of coatings obtained after 30 min deposition in an electrolyte, containing (g/L): Cu^{2+} - 1, Zn^{2+} - 50 and H_2SO_4 - 130 at 1 A/dm².

Fig. 1b. SEM micrographs of coatings obtained after 30 min deposition in an electrolyte, containing (g/L): Cu^{2+} - 1, Zn^{2+} - 50 and H_2SO_4 - 130 at 5 A/dm².



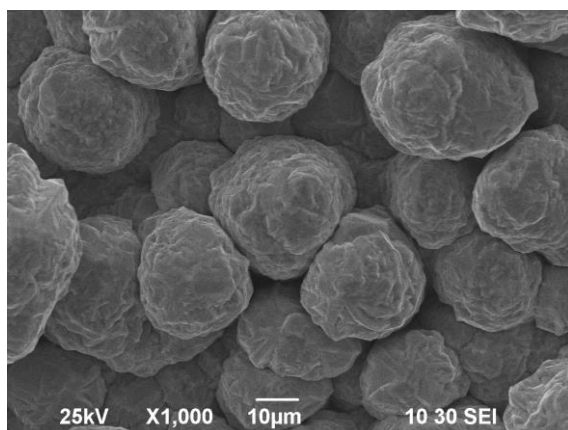
(a)



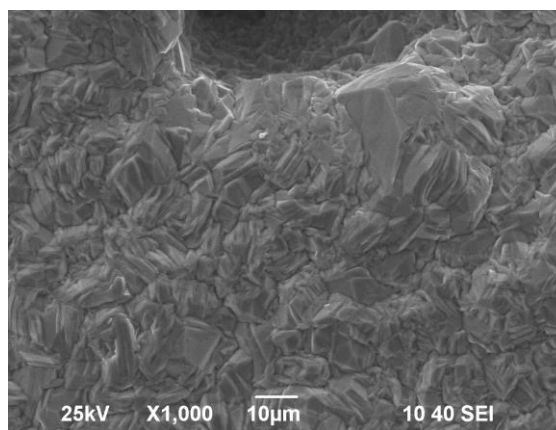
(b)

Figure 2a. SEM micrographs of coatings obtained after 30 min deposition in an electrolyte, containing (g/L): Cu^{2+} - 10, Zn^{2+} - 50 and H_2SO_4 - 130 at 1 A/dm².

Figure 2b. SEM micrographs of coatings obtained after 30 min deposition in an electrolyte, containing (g/L): Cu^{2+} - 10, Zn^{2+} - 50 and H_2SO_4 - 130 at 5 A A/dm².



(a)



(b)

Figure 3a. SEM micrographs of coatings obtained after 5 h deposition in an electrolyte, containing (g/L): Cu^{2+} - 10, Zn^{2+} - 50 and H_2SO_4 - 130 at 1 A/dm².

Figure 3b. SEM micrographs of coatings obtained after 5 h deposition in an electrolyte, containing (g/L): Cu^{2+} - 20, Zn^{2+} - 50 and H_2SO_4 - 130 at 1 A/dm².

deposition at current density 1 A/dm² and potentials between -0.430 and -0.530 V vs SSE in electrolytes containing (g/L) Cu²⁺ - 10 or 20 and H₂SO₄ - 130. The coating obtained in the electrolyte containing 10 g/L Cu²⁺ is dark red, rough and brittle (Figure 3a). The coating obtained in the electrolyte, containing 20 g/L Cu²⁺ is light red, smooth and semi-bright (Figure 3b). Light red, smooth and semi-bright coatings of pure Cu deposit are obtained at current density 0.5 A/dm² in electrolytes containing 10 or 20 g/L Cu²⁺. The current yield of Cu deposition is 97 – 99% in all cases.

Electroextraction of Cu in the presence of Fe²⁺ ions.

Figure 4 shows the dependence of current efficiency of Cu deposition (CE_{Cu}) on Fe²⁺ concentration. It is seen that CE_{Cu} sharply decreases with the increase in Fe²⁺ concentration during deposition in electrolytes containing 10 g/L Cu²⁺ and 130 g/L H₂SO₄ at a current density of 2 A/dm² (curve 1) and in electrolytes containing 50 g/L Cu²⁺ and 130 g/L H₂SO₄ at a current density of 1 A/dm² (curve 2). It is higher than 90% and is practically independent on Fe²⁺ concentration during deposition in electrolytes containing 50 g/L Cu²⁺ and 130 g/L H₂SO₄ at a current density of 2 A/dm² (curve 3). The very weak influence of Fe²⁺ on the electroextraction of Cu from electrolytes containing 50 g/L Cu²⁺ can be explained by the impeded access to the cathode of the Fe³⁺ ions formed on the anodes [23]. The reduction of ferric ions to ferrous ions on the cathode and the increased H₂ evolution may lead to a decrease in Cu current efficiency. This is, probably, the reason for the significantly

lower current efficiency when the concentrations of Cu²⁺ and Fe²⁺ are equal (10 g/L).

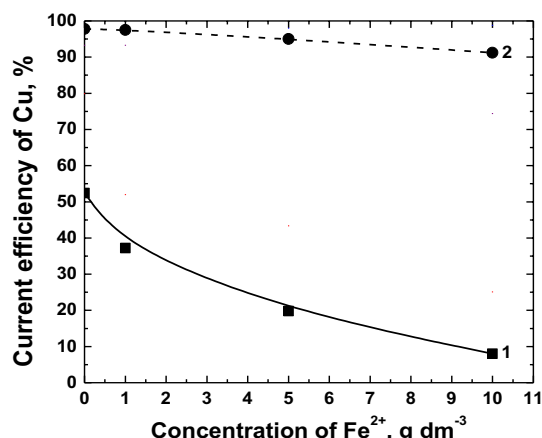
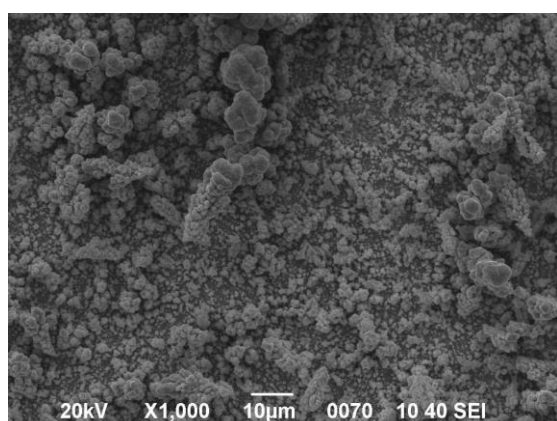
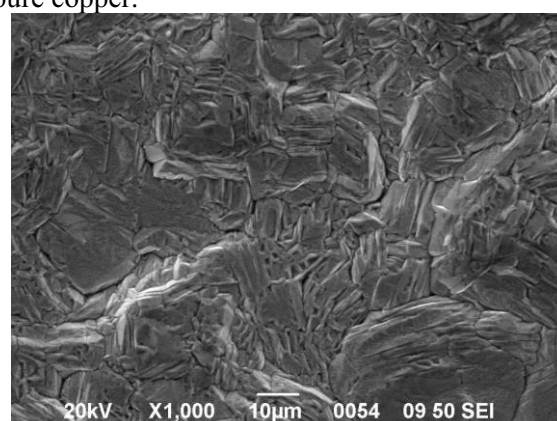


Figure 4. Dependence of the current efficiency of 5 hours galvanostatic deposition of copper as a function of Fe²⁺ concentration. Electrolytes: 1) 10 g/L Cu²⁺ and 130 g/L H₂SO₄, current density 2A/dm²; 2) 50 g/L Cu²⁺ and 130 g/L H₂SO₄, current density 1 A/dm²; 3) 50 g/L Cu²⁺ and 130 g/L H₂SO₄, current density 2 A/dm².

Figures 5a and b present SEM micrographs of copper coatings deposited for 5 h at 2 A/dm² in electrolytes containing 10 g/L Cu²⁺, 10 g/L Fe²⁺ and 130 g/L H₂SO₄ (Figure 5a) and 50 g/L Cu²⁺, 10 g/L Fe²⁺ and 130 g/L H₂SO₄ (Figure 5b). It is observed that coatings obtained from electrolytes containing 10 g/L Cu²⁺ in the presence of Fe²⁺ ions are dark red, rough and mat, probably, as a result of increased hydrogen evolution. The coatings obtained from electrolytes containing 50 g/L Cu²⁺ are light red, smooth and bright. The results of the microprobe analysis indicate that all coatings are of pure copper.



(a)



(b)

Figure 5a. SEM micrograph of Cu coating obtained in an electrolyte containing 10 g dm⁻³ Cu²⁺, 10 g dm⁻³ Fe²⁺ and 130 g dm⁻³ H₂SO₄. Current density 2A A/dm². Potential -0.615 V. Deposition time 5 h.

Figure 5b. SEM micrograph of Cu coating, obtained in an electrolyte, containing Cu²⁺ - 50 g dm⁻³, Fe²⁺ - 10 g dm⁻³ and H₂SO₄ - 130 g dm⁻³. Current density 2A/dm². Deposition time 5 h.

Electroextraction of Cu in the simultaneous presence of Zn²⁺ and Fe²⁺ ions.

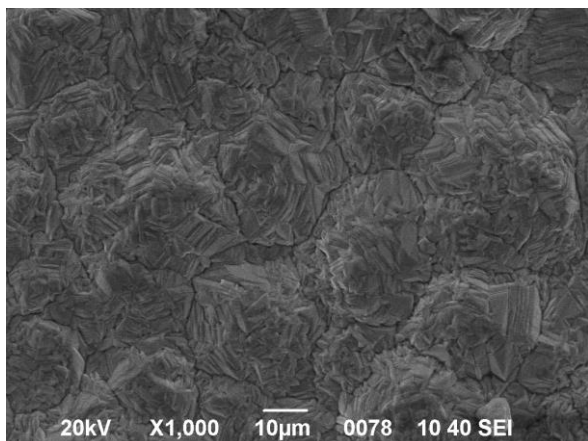


Figure 6. SEM micrograph of Cu coating, obtained in electrolytes, containing (g/L): Cu²⁺ - 50, Zn²⁺ - 50, Fe²⁺ - 10 and H₂SO₄ - 130. Current density 2 A/dm². Deposition time 5 h.

Figure 6 show deposit composition, surface appearance and SEM images of a copper coating deposited for 5 h at 2 A/dm² current density in an electrolyte containing 50 g/L Cu²⁺, 50 g/L Zn²⁺, 10 g/L Fe²⁺ and 130 g/L H₂SO₄. It is seen that the simultaneous presence of Zn²⁺ and Fe²⁺ ions leads to obtaining of a fine-grained coating similar to the coating obtained in the electrolyte containing only 50 g/L Cu²⁺ and 10 g/L Fe²⁺ (Figure 5b). The coating is light red in color, smooth and semi-bright. The microprobe analysis evidences that the coating is of pure copper.

CONCLUSION

Depending on the ratio between Cu²⁺ and Zn²⁺ ions in the electrolyte, conditions can be created favoring deposition of pure Cu and Zn phases, as well as an alloyed Cu-Zn phase with a different composition.

The coatings produced by means of galvanostatic deposition from electrolytes containing 1 g/L Cu²⁺, 50 g/L Zn²⁺ and 130 g/L H₂SO₄ are composed predominantly of Zn (80-90%). The coatings are black, smooth and powdery. Pure Cu coating is produced by deposition at 1 A/dm² in an electrolyte containing 10 g/L Cu²⁺, 50 g/L Zn²⁺ and 130 g/L H₂SO₄. Alloyed Cu-Zn coatings are produced at 5 A/dm² with Zn content of 4.9%. The coating deposited at 1 A/dm² is orange in color, smooth and semi-bright, while the coating obtained at 5 A/dm² is dark red, spongy and brittle.

To produce fine-grain, dense and smooth coatings of pure Cu in the presence of Zn²⁺ ions, the concentration of Cu²⁺ ions in the electrolyte should

be higher than 10 g/L and the current density should not exceed 1 A/dm².

On galvanostatic Cu deposition at 2 A/dm² from electrolytes containing 10 g/L Cu²⁺ and 130 g/L H₂SO₄, the current efficiency abruptly declines with the increase in Fe²⁺ concentration from 1 to 10 g/L. The coatings obtained at a current density of 2 A/dm² from electrolytes containing 10 g/L Cu²⁺ and 10 g/L Fe²⁺ are of pure copper, dark red in color, brittle and powdery.

The current efficiency of copper electrodeposition in electrolytes containing 50 g/L Cu²⁺ and 130 g/L H₂SO₄ is higher than 90% and slightly decreases with the increase in Fe²⁺ concentration. The coatings are of pure copper, light red in color, smooth and semi-bright in appearance.

On galvanostatic deposition at 2 A/dm² from electrolytes containing 50 g/L Cu²⁺, 50 g/L Zn²⁺, 10 g/L Fe²⁺ and 130 g/L H₂SO₄, the obtained coatings are of pure copper, light red in color, smooth and semi-bright.

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ИЗВЛИЧАНЕ НА МЕТАЛИ ОТ ТВЪРДИ МЕТАЛУРГИЧНИ ОТПАДЪЦИ.
ГАЛВАНОСТАТИЧНА ЕЛЕКТРОЕКСТРАКЦИЯ НА МЕД ОТ СУЛФАТНИ
ЕЛЕКТРОЛИТИ, СЪДЪРЖАЩИ Zn^{2+} И Fe^{2+} ЙОНИ

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(Резюме)

Посредством галваностатични методи е изследвана електроекстракцията на мед от сернокисели (130 g/L) сулфатни електролити в присъствието на Zn^{2+} или Fe^{2+} йони. Покритията, отложени от електролити, съдържащи 1 g/L Cu^{2+} и 50 g/L Zn^{2+} са съставени предимно от Zn (80-90%). Покритията са черни и прахообразни. Покрития от чиста мед се получават при 1 A/dm² от електролити, съдържащи 10 g/L Cu^{2+} и 50 g/L Zn^{2+} . Покритията са светлочервени на цвят, гладки и полублестящи. Добивът по ток на медното отлагане при 2 A/dm² от електролити, съдържащи 10 g/L Cu^{2+} рязко намалява с нарастването на концентрацията на Fe^{2+} . Добивът по ток е по-висок от 90% и практически не зависи от концентрацията на Fe^{2+} при отлагане от електролити, съдържащи 50 g/L Cu^{2+} . Покритията, получени от електролити, съдържащи 10 g/L Cu^{2+} в присъствието на Fe^{2+} йони са тъмnochервени, грапави и матови, а тези получени от електролити, съдържащи 50 g/L Cu^{2+} са светлочервени, гладки и блестящи. И в двата случая покритията са от чиста мед.