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Technology improvement of copper plating on the constituents of internal crusher gauges

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The copper is electroplated on the external surface of the constituents of internal crusher gauges (UK-3 and UK-4). The basic material of the constituents for internal crusher gauges is stainless steel (Č. 4770), and copper is plated on the strike. According to the previously applied technology, copper deposits obtained from the cyanide solution were used for the strike. Regarding strict ecological regulations for using cyanide solutions in plating shops, a new technology consisting of the electrolytic activation of the basic material surface and the electrodeposition of a thin nickel coating from the chloric solution is proposed. The possibility to apply this improved technology of copper plating on the constituents of internal crusher gauges was examined in the paper. The results of the effects of the input process parameters on the properties of deposits and on the electrodeposition rate were reported. Optimum conditions of copper plating on the constituents of internal crusher gauges were proposed.

Key words: copper plating technology, internal crusher gauges, copper electrolytic coating, stainless steel, deposit properties, electrodeposition rate

Introduction

A functional requirement for the constituents of internal crusher gauges is the protection of external surfaces with copper electrolytic coatings (UK-3 and UK-4). The copper coating on the constituents has a required thickness from 650 to 750 μm . In order to get a required thickness of the copper coating after mechanical treatment, the average copper coating thickness during electrodeposition, should be from 1.2 to 1.5 mm. The distribution of the copper coating thickness on the surface of constituents depends on electrolyte chemical composition and concentration, current regime, type of accommodation of constituents in jigs, electrolyte stirring and position of the jigs and the anodes in the bath [1,2].

The basic metal of crusher constituents is high chromium steel which has undergone heat treatment before copper plating. Coating electrodeposition on stainless steel is difficult because the surface of metals is coated with oxides, thus being passivated. Heat-treated stainless steel has products on its surface, as a result of processes at high temperatures (most often oxides). These products strongly adhere to the basic metal and make the preparation of the surface difficult for coating electrodeposition [3].

Coatings of copper are electrodeposited on a basis metal or on a strike, depending on the nature of a basic metal and the function of a coating. The copper coating can be a strike for other coating electrodeposition. If the copper coating is electrodeposited on steel, then the first thin copper coating from the cyanide solution is electrodeposited. Later, after rinsing, the electrodeposition of copper from the acid electrolyte continues [4].

Nickel as a strike can be used in the process of copper plating on steel. For nickel electrodeposition on stainless

steel (Č.4770 and similar), different electrolytes and technological treatments are recommended in the literature. Prior etching of the basic metal and later electrodeposition of a thin nickel strike from the solution of nickel chloride and hydrochloric acid gave good results. The etching of steel Č.4770, chemical and electrochemical, can be practiced. After these procedures, a clean metal surface is ready for the short-duration electrodeposition of the nickel coating (nickel 1) [5].

After profuse rinsing, the second coating of nickel from the sulphuric-chloride electrolyte is electrodeposited on the constituents. The coating of nickel electrodeposited in this electrolyte (nickel 2) should not be porous. The compact nickel coating enables later copper plating from the sulphuric electrolyte without immersion plating. Therefore the total system of coating (nickel 1- nickel 2 – copper) has good adhesion on the basic metal of crusher constituents [6,7].

In accordance with recently applied technology, the preparation for copper plating on the constituents of internal crusher gauges included: the electrodeposition of the copper strike from the cyanide electrolyte (Kuprodigresiv) Chemical plant (CP Podnart), the strike of nickel from the electrolyte "Nickel BT-EXSTRA 7630" CP Podnart and the copper coating from the sulphuric acid electrolyte. In the preparation of the crusher basic metal (Č.4770) detergent degreasing was used, although insufficient data has been reported. The protection of the interior surface, where coatings should not be electrodeposited, was carried out using resin. This represents the intricate single protection [1,8].

The purpose of this work is to examine the possibility to use the proposed technology of copper plating with the strike of nickel 1- nickel 2 on the constituents of internal crusher gauges and to define the conditions of processes when coatings with required quantity are deposited.

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Theoretical part

Copper and electrolytes for copper electrodeposition

Copper is a metal with extensive use, as a single metal or in the form of alloys. Copper has a high melting point of 1083 °C and a density of 8.9 g dm⁻³. Copper in compounds occurs as Cu (I)-cuprous ions and Cu(II)-cupric ions, and the electrochemical equivalent for Cu(I) is 2.372 g/Ah, and for Cu(II) is 1.186 g/Ah. The standard electrode potentials of copper are:

$$E_{\text{Cu,Cu}^+} = +0.52 \text{ V}, E_{\text{Cu,Cu}^{2+}} = +0.34 \text{ V}, E_{\text{Cu}^+, \text{Cu}^{+2}} = +0.17 \text{ V}$$

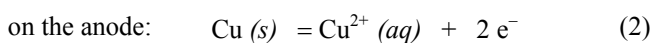
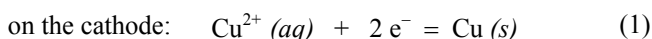
The standard electrode potential for copper has a higher value than hydrogen; therefore, copper is deposited before hydrogen on the cathode from acid solutions. Copper does not preserve metal surface for a long period, because in the presence of the compounds of sulphur it is quickly coated which the black copper-sulphide. The copper-oxide coating is obtained in humid atmosphere, while in the presence of humidity and carbon dioxide it is coated with green basic salts of copper [4].

Acid and alkaline electrolytes are used for copper plating. The most frequently used alkaline electrolyte is cyanide electrolyte and the most frequently used acid electrolyte is sulphuric electrolyte. For copper plating direct and reversing current regimes are used [9,10].

From cyanide solutions, copper is reduced on the cathode from the univalent copper ion. The main compounds of the cyanide electrolyte are the complex salt and free cyanides. Due to low dissociation of the cyanide complex, the concentration of the copper ions is small so the potential Cu(I) is very negative, from -0.9 to -1.5 V. Thus the copper potential approaches the iron potential, therefore iron do not immersion plating from the cyanide solution. Copper can be thus electrodeposited from the cyanide electrolyte on steel. Together with copper on the cathode there is hydrogen evolving. When the basic metal is steel, then the penetration of hydrogen through the crystal lattice makes brittle. The alkaline cyanide electrolytes change due to the absorption of carbon dioxide from the air. The cyanide electrolytes are more sensitive than other electrolytes to the presence of additives [11].

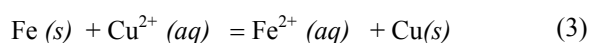
The acid electrolytes are based on Cu(II) ions, because Cu(I) ions are unstable in the air. The sulphuric electrolyte has the widest use of all acid electrolytes and its basic components are: copper sulphate and sulphuric acid. The sulphuric acid is added in order to increase electrolytic conduction and to prevent to the forming of basic salts on the cathode and as well as to reduce the forming of copper(I) ions and their hydrolisis. This electrolyte is cheap, stable, allows usage of major current density and current efficiency achieved is to 100 %.

The reactions on the electrodes in the acid electrolytes are:



The faults of the sulphuric electrolyte are:

- large grain crystal structure of coatings,
- low distribution of metals,
- copper coating cannot be electrodeposited directly on steel because of immersion plating:



The effect of a small amount of organic additives in sulphuric electrolyte is compact copper coatings with a fine grain crystal structure. The work current of cathode density can be considerably increased by stirring the electrolyte with the pure compressed gas and by moving the cathode. Also, the use of current pulsating regimes and the large current density affects the improving of coating quality [2,11].

The anodes are always made of electrolytic copper, cast or rolled. The copper anodes can contain additions, e.g. copper anodes, deoxidized with phosphorus, contain up to 0.005 % of phosphorus. During work, a black hard-adhering film is formed, which does not require increased tank voltage for electrolysis. These anodes do not make anode silt. Potassium sodium tartarate is added into the electrolyte, in order to achieve easier dissolution of anodes, since tartarate ions make complex ions with copper.

The copper coating, according to the requirements for the protection of crusher constituents, needs to be compact, to have good adhesion on the nickel strike, and to tolerate mechanical treatment. After mechanical treatment the copper coating should have a thickness from 0.6 to 0.9 mm, depending on the constituents that are protected.

The technology improvement of the protection of crusher constituents

The technology improvement of the surface protection of crusher constituents with a three-coating system includes the following treatments:

1. degreasing with solvent (acetone or trichloroethylene),
2. degreasing in alkaline solution (heat chemical and electrochemical),
3. rinsing in running water,
4. chemical etching (in a sulphuric acid solution or an acid compound),
5. rinsing in running water,
6. activation of surface and electrodeposition of the first nickel strike (electrolyte 1),
7. rinsing in running water,
8. electrodeposition of the second nickel strike (electrolyte 2),
9. rinsing in running water,
10. copper plating,
11. rinsing in running water,
12. drying,
13. grinding and
14. control of coating condition.

Experimental part

The specimens are the crusher constituents: body and top made of stainless steel Č.4770 (the chemical composition is in Table 1):

Table 1. The chemical composition of steel Č.4770 [w.%]

C	Si	Mn	Cr	Mo	Fe
0.5	0.5	1	14	0.4	up to 100

Dimensions of the crusher constituents are:

- the crusher body: ϕ 15.3x21.8 mm (ϕ 17x21.8 mm),
- the crusher top: ϕ 15.3x3.4 mm (ϕ 17x4.2 mm).

The drawings of the bodies and the tops are given in [1].

The experimental work in laboratory included the investigation of surface preparation, activation and electrodeposition of the first nickel strike on the basic metal of the crusher constituents (Č.4770). The electrodeposition of nic-

kel was the first planned on the mechanical by treated, crusher constituents and then on the crusher constituents heat treated according to the technological procedure [1]. On the heat treated constituents, the emerged products are the first mechanically cleaned and then chemically treated. If the electrodeposited coating strike of nickel 1 is found to have good adhesion on the basic metal and that it is compact, then the investigation of the second nickel strike electrodeposition from the sulphuric electrolyte is continued, followed by the electrodeposition of the copper coating from the sulphuric electrolyte.

According to the proposed technology procedure for the basic metal preparation, it is necessary to prepare solutions with specific chemical compositions and concentrations given in Table 2:

Table 2. Compositions and concentrations of the solutions for the preparation of the basic material surface

No	Name of chemicals	Concentration [gdm ⁻³] or [mldm ⁻³]			
		c	Solution for alkaline cleaning	Solution for acid cleaning I	Solution for acid cleaning II
1.	Sodium hydroxide, NaOH	gdm ⁻³	30		
2.	Sodium carbonate, Na ₂ CO ₃	gdm ⁻³	25		
3.	Sodium orthophosphate Na ₃ PO ₄	gdm ⁻³	50		
4.	Sulphuric acid, H ₂ SO ₄ (ρ = 1.84 gcm ⁻³)	mldm ⁻³		110	60
5.	Nitric acid, HNO ₃ (ρ = 1.4 gcm ⁻³)	mldm ⁻³			40
6.	Hydrochloric acid, HCl (ρ = 1,18 4 gcm ⁻³)	mldm ⁻³			40

It was planned to investigate the protection of the surface on the crusher constituents where coatings are not be electrodeposited, using lacquer, resins or teflon when the protection of interior surfaces is in question. The electrodeposition of coatings was carried out on the jig with one constituent.

According to the proposed technology for the electrochemical activation of the basic metal and the electrodeposition of the nickel 1 coating, electrolyte 1 (Table 3) and electrolyte 2 (Table 3) for the electrodeposition of nickel 2 are used.

Table 3. Compositions and concentrations of the solutions for the basic metal activation and for the electrodeposition of nickel 1 and nickel 2 coatings

No	Name of chemicals	Concentration [gdm ⁻³] or [mldm ⁻³]		
		c	Solution 1	Solution 2
1.	Nickel-chloridehexahydrate, NiCl ₂ 6H ₂ O	gdm ⁻³	250	40
2.	Hydrochloric acid, HCl (ρ = 1.18 4 gcm ⁻³)	mldm ⁻³	100	
3.	Nickel-sulfatehexahydrate, NiSO ₄ 6H ₂ O	gdm ⁻³		240
4.	Boric acid, H ₃ BO ₃	gdm ⁻³		30
5.	Saccharin, C ₆ H ₄ SO ₂ NHCO	gdm ⁻³		0.9-1.2
6.	Formaldehyde, HCHO	mldm ⁻³		1.0-1.2

Note: For the electrodeposition of nickel 2 the pH values are from 4.5 to 5.5

For the copper electrodeposition of greater thickness, the sulphuric acid electrolyte is proposed. In this procedure the

electrolyte of following chemical composition and concentration was used:

Copper sulphate, CuSO ₄ 5 H ₂ O	from 210 to 250 g dm ⁻³
Sulphuric acid, H ₂ SO ₄ (ρ = 1.84 gcm ⁻³)	from 40 to 55 g dm ⁻³
Potassium sodium tartarate, KNaC ₄ H ₄ O ₆	2 g dm ⁻³

The preparation technology of the crusher constituents basic metal surface and the electrodeposition of the three system of coating (nickel 1- nickel 2- copper) was carried out with process parameters values given in Table 4.

The electrodeposition of coatings is carried out on a single constituent, placed vertically in the bath in a system of plane parallel plates (a specimen with the jig is placed between two parallel anodes).

The copper electrodeposition is carried out using the cathode displacement and the stirring of electrolyte with compressed gas.

If the copper electrodeposition procedure is interrupted after 6 to 8 hours, before reelectrodeposition the constituents are instantly etched in the 20% sulphuric acid solution. After that, they are rinsed and placed in the bath under current.

Table 4. Values of the basic parameters for chemical and electrochemical processes

	Name of proceses	Parameters of processes			
		Temperature [°C]	Density of current [A dm ²]	Voltage [V]	Time of treatment [s]
1.	Chemical degreasing	60			300
2.	Electrochemical degreasing	60	5	1-2	300
3.*	Chemical etching I	50-60			60
4.*	Chemical etching II	20-25			60-180
5.	Electrochemical activation of surface	20-25	5-10	1.2	120-180
6.	Electrodeposition of nickel 1	20-25	5	1.2	300
7.	Electrodeposition of nickel 2	45-50	5	1.2	420
8.	Electrodeposition of copper	22-25	1-10	0.6-1.5	3600 and more

Note * Chemical etching I is done in solution 1, and chemical etching II in solution 2.
Flourinsing in water during 300 s is done after each chemical and electrochemical process.

After copper electrodeposition, the constituents were well rinsed, then dried and taken down from the jigs. After the examination of the copper coating appearance, mechanical treatment was carried out in to obtain the required dimensions.

During the procedure of crusher constituents protection in the presented technology defects can occur. Defects can appear because of insufficient preparation of the basic metal surface, bad surface activation or value deviation of process parameters of the nickel 1, nickel 2 or copper coating electrodeposition.

New technology application results in the crusher constituents protection

According to the experimentation plan, after mechanical and heat treatment, crusher constituents were taken. Tools were made for single electrodeposition.

Solutions for chemical and electrochemical treatment processes were prepared. Chemical analyses of the concentration of solution basic components were performed.

The examination of the required basic metal preparation and the first nickel coating electrodeposition was carried out according to the proposed technology treatment and the conditions given in Table 2.

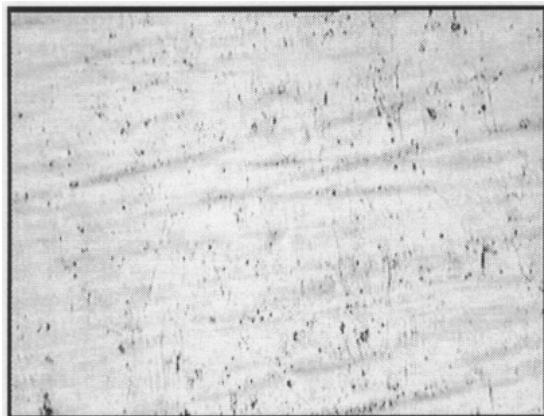
The heat-treated products adhered firmly to the basic metal. The crusher constituent surfaces coated by heat-treated products were treated with steel brushes, then degreased in the alkaline solution and treated in the solution of acids. A clean metal surface was obtained.

The examination of the constituent surfaces protected with the nickel 1 coating showed a compact grey nickel coating, well adhering to the basic metal. The nickel from the sulphuric electrolyte (nickel 2) was electrodeposited on nickel 1. A brilliant and compact coating was obtained with good adhesion on nickel 1. The copper electrodeposition on nickel 2, during one hour, gave a brilliant compact metallic coating. The copper coating has good adhesion on nickel 2.

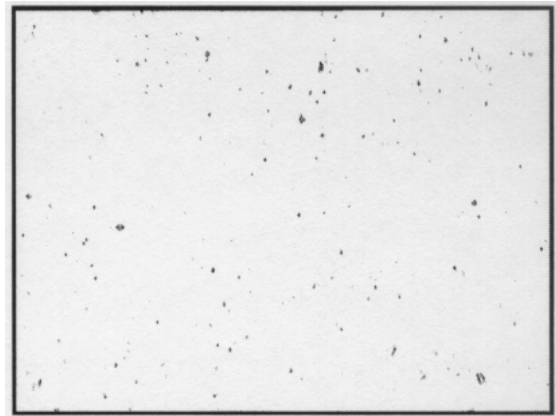
It was established that resin (used for interior surface protection) becomes soft during the preparation process, and does not protect efficiently. Also, resin makes rinsing difficult. In further experimental work teflon was applied. The use of teflon screen protection is efficient for the protection of surfaces where coatings should not be electrodeposited.

In Figs.1a, 1b and 1c, the morphologies of nickel 1, nickel 2 and copper coatings were presented. On some specimens, due to the deviations from the given electrodeposition conditions, the compact coating of nickel 2 was not electrodeposited, which caused copper immersion plating at places with pores with the morphology presented in Fig.1d.

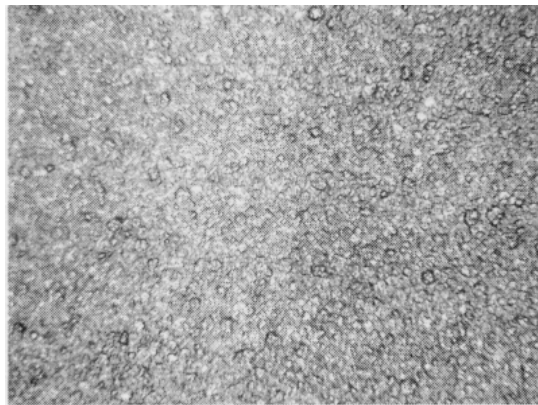
The copper coatings were electrodeposited with a current density from 1 to 10 A dm⁻², at a solution temperature from 22 to 24°C, using the cathode displacement and the electrolyte stirring with compressed gas. Copper coatings were electrodeposited to a thickness of about 1500 µm. The copper electrodeposition on the specimens was carried out continually or with stopping after 6 hours. The investigation results showed that for the copper electrodeposition from the sulphuric electrolyte in the given conditions (at room temperature, using the electrolyte stirring with compressed gas and the cathode displacement) maximum current densities up to 5 A dm⁻² can be used. A fine-grain structure of the copper coating, thus obtained, can be mechanically treated. The duration of the copper coating electrodeposition of a thickness of about 1500 µm (within the given conditions) is 46 hours. The morphology of such copper coating is presented in Fig.2a. The edge effect occurs on the lower edge (the edge near the bath bottom) if copper is electrodeposited vertically on the crusher body. Due to, this rough deposit, difficult to be treated, is formed. Its morphology taken by optical microscopy is presented in Fig.2b.



a)



b)

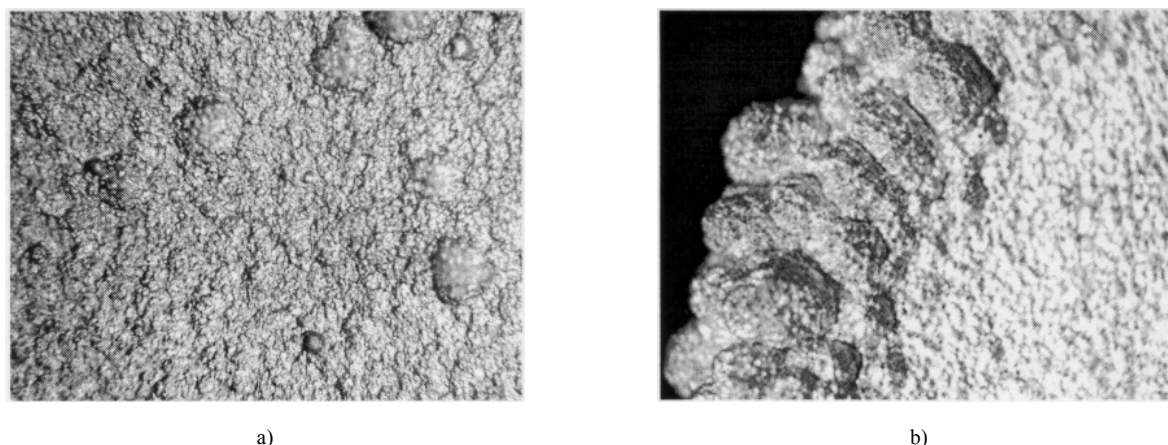


c)



d)

Figure 1. Morphology of the deposit obtained by optical microscopy, magnification 300x, a-nickel 1, b- nickel 2, c- copper and d- copper partly immersion plated.



a)

b)

Figure 2. Morphology of the copper coating electrodeposited from the sulphuric electrolyte with a current density of 5 A dm^{-2} : a- on the specimen surface, magnification 300x, b- on the specimen edge, magnification 50x.

Table 5. Results of the measurement of the coating thickness on the crusher body (initial dimension: $\phi 16,51 \text{ mm}$) with the copper coating electrodeposited for 46 hours

Height of measuring on the crusher body "0" (edge near the bottom of the tank) [mm]	The diameter [mm]			The total coating thickness [μm]			
	point 1	point 2	point 3	point 1	point 2	point 3	the average thickness
19.5	19.43	19.73	19.66	1 460	1 610	1 570	1 540
10.0	19.54	19.72	19.64	1 510	1 600	1 570	1 560
5	20.97	21.39	21.69	2 230	2 440	2 570	2 410

Note: Points 1,2 and 3 were distributed along the diameter under the angle of 120° . The total thickness includes the thicknesses of nickel 1, nickel 2 and copper, but since the thickness of nickel 1 and nickel 2 is up to $10\mu\text{m}$, which is neglectable in relation to the thickness of copper, the discussion treats the oval copper coating.

Because copper is electrodeposited vertically on the crusher constituents in a system of plane parallel anodes, the oval shape of the constituent cross section can be noticed. The vertical cross section shows a pronounced edge effect, especially on the lower edge near the bath bottom, as presented in Table 5.

By increasing the copper electrodeposition current density to 10 A dm^{-2} (using the cathode and displacement the electrolyte stirring) a coating thickness of $1500 \mu\text{m}$ is obtained with the electrodeposition time of 21 hours. However, grains are considerably increased and is dendrite structure formed. Grinding of that coating structure reveals the recesses on the metal surface. That shows that a granule formed above these recesses had the effect of a screen, which did not allow further copper electrodeposition in the "shadow" under it. Granule tops dimensional surplus, are taken down by mechanical treatment uncovering the places where the copper crystallization process was interrupted.

For the copper electrodeposition with only the cathode displacement, the use of current density from 1 to 2 A dm^{-2} is recommended. Copper coatings electrodeposited under these conditions have a fine structure. Which gives smooth appearance after the mechanical treatment. In order to obtain copper coatings of thicknesses of about $1500 \mu\text{m}$, electrodeposition time is from 84 to 120 hours.

Conclusion

The analysis of the technology for the protection of internal crusher constituents is performed. Regarding modern treating possibilities and existing ecological regulations, the change of the previously applied technology, was proposed. In other words, the activation surface treatment and electrodeposition of thin nickel coatings from the acid electrolyte

is proposed, substituting copper coatings electrodeposited from the cyanide electrolyte.

The examination of crusher constituents protection possibility, regarding a new technology of the three-coating system (nickel 1- nickel 2- copper), was performed. The technology of basic metal surface preparation (Č.4770) was developed, with heat treatment preceding surface protection.

After mechanical, chemical and electrochemical treatment of the basic metal, a clean metal surface was obtained and then nickel 1 was electrodeposited on it.

The nickel 1 coating replaced the copper coating electrodeposited from the cyanide electrolyte. The nickel 1 coating is very compact, opaque-gray and has good adhesion on the basic metal. The white-brilliant nickel 2 coating is electrodeposited on nickel 1 and has good adhesion on nickel 1. The copper coating is electrodeposited on nickel 2 under different conditions, and it was established that it had good adhesion on nickel 2.

The copper electrodeposition process and the change of coating properties were monitored regarding the change of process parameters. The copper coating with a thickness of $1500 \mu\text{m}$ from the sulphuric electrolyte was electrodeposited in 46 hours. The current density was 5 A dm^{-2} and the cathode displacement and the electrolyte stirring with compressed gas were applied. Such coating has a structure suitable for mechanical treatment. With the current density increasing up to 10 A dm^{-2} dendritic structure is formed, which is hard to treat mechanically. By reducing the current density from 1 to 2 A dm^{-2} , a compact coating with fine structure was obtained, but electrodeposition time increased considerably (up to 120 hours).

The protection of the crusher constituents surface where coatings should not be electrodeposited was successfully obtained with lacquer and teflon screens.

The proposed technology improvement for the protection of internal crusher constituents improved the protection with the three-coating system as well as the ecological conditions in the premises and laboratory.

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Poboljšanje tehnološkog postupka elektrolitičkog taloženja bakra na elementima unutrašnjih krešera

Bakar se elektrolitički taloži na spoljne površine elemenata (telo i poklopac) unutrašnjih krešera (UK-3 i UK-4). Osnovni metal elemenata unutrašnjih krešera je Č.4770, zbog čega se bakar taloži na podsloj. Prema do sada primenivanoj tehnologiji za podsloj korišćena je prevlaka bakra dobijena iz cijanidnog elektrolita. Uzimajući u obzir sve strožije ekološke propise pri upotrebi cijanidnih rastvora u galvanizacijama predložen je poboljšani tehnološki postupak, koji umesto podsloja bakra uključuje elektrohemijsko aktiviranje površine osnovnog metala i taloženje tankog sloja nikla iz hloridnog elektrolita. U ovom radu je ispitivana mogućnost primene predloženog novog postupka taloženja bakra na elementima krešera. Prikazani su rezultati ispitivanja uticaja pojedinih parametara procesa na svojstva prevlaka i na brzinu taloženja. Predloženi su optimalni uslovi za taloženje prevlaka bakra na elementima krešera.

Gljučne reči: tehnološki postupak, krešeri, elektrolitička prevlaka bakra, prokron, svojstva prevlaka, brzina taloženja.

Amélioration du procédé technologique de placage électrolytique du cuivre sur les éléments de blocs crusher intérieurs

Le cuivre forme le dépôt électrolytique sur les surfaces extérieures des éléments de bloc crusher intérieurs (UK-3 et UK-4). Le métal de base des éléments de blocs crusher intérieurs (cylindre et bouchon) est l'acier inoxydable (Č. 4770) et le cuivre est précipité sur la sous-couche. Selon la technologie appliquée auparavant, les dépôts de cuivre pour la sous-couche étaient obtenus de l'électrolyte de cyanure. Etant donné les règles écologiques (concernant l'application de l'électrolyte de cyanure pour la galvanisation) de plus en plus sévères, on a proposé un procédé technologique amélioré dans lequel la surface du métal de base est activée électrolytiquement et la couche mince du nickel est électrodéposée de l'électrolyte de chlorure. Ce papier traite la possibilité d'utiliser cette technologie améliorée de placage électrolytique du cuivre sur les éléments de blocs crusher. Les résultats de l'investigation des effets de quelques paramètres de processus sur les propriétés des dépôts et la vitesse d'électrodéposition sont présentés. Les conditions optimales pour le placage électrolytique du cuivre sur les éléments de blocs crusher sont également proposées.

Mots-clés: procédé technologique, crushers, dépôt électrolytique de cuivre, acier inoxydable, propriétés du dépôt, vitesse d'électrodéposition.