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Қ. И. Сәтпаев атындағы Қазақ ұлттық техникалық зерттеу университеті

# Х А Б А Р Л А Р Ы

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## ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК  
РЕСПУБЛИКИ КАЗАХСТАН  
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## NEWS

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**COPPER POWDERS FORMATION IN THE CATHODIC  
AND ANODIC HALF-PERIODS  
WITH INDUSTRIAL ALTERNATING CURRENT**

**Abstract.** For the first time, an installation was created for producing copper powders in the anodic and cathodic half-periods of industrial alternating current with a frequency of 50 Hz. This device consists of two electrolytic cells interconnected in parallel, in each electrolyzer there are two copper electrodes. Two diodes are also included in the electrochemical circuit to allow current flowing in opposite directions. The influence of electrodes current density, sulfuric acid concentration, copper ions, on current output of copper powder formation in each cell was investigated. It was established that the current output increases within the current density from 2000-8000 A/m<sup>2</sup> and decreases with further increase of this parameter. When sulfuric acid concentration was 100 g/l and copper ions concentration was 15 g/l, the maximum current output of powder in electrolyzes was 50.1-53.3%, and the total current output exceeded 100%.

Using a JSM-661 LV scanning electron microscope, the shape and size of obtained powders were investigated. On the basis of the conducted studies and captured oscillograms, the mechanism for copper powders formation in the anodic and cathodic half-cycles of alternating current was established. For the first time it was shown that the proposed installation allows to obtain copper powders in two half periods of alternating current. It was established that at the same time ultrafine metal powders with particle sizes of 1.0-1.5 microns had been formed.

**Keywords:** alternating current, copper powders, titanium, diode, electrode, electrolyte, cathode and anodic half-periods, oscillogram.

Electrochemical reactions mechanism, which usually occurs in the electrodes, is very complex. Electrochemical reactions course depends on an electrolyte composition and its temperature, current density in the electrode and their nature, hydrodynamic conditions in the electrolyte and other parameters [1]. For example, when the concentration of copper (II) ions is high and the current density at the cathode is not higher than one electrode surface, a layer of copper is formed. It is known that at a low concentration of metal ions, and a high surface density of the cathode (i.e., in case of exceeding the limiting current densities), copper (II) ions are restored and precipitate to form small powder particles [2].

In recent years, many scientific studies have been carried out to obtain ultrafine and nano-sized copper powders [3-15].

With polarization using unsteady current, the nature and nature of the reactions that take place on the cathode surface cannot be fully determined. Our previous studies have shown the possibility of obtaining various metal compounds with polarization by industrial alternating current with a frequency of 50 Hz, and also demonstrated the ability to manufacture dispersed copper powders [16, 17]. In our earlier studies, a pair of titanium electrode with a small surface area and a large-area copper electrode was collected for the first time, and the possibility of producing ultrafine copper powders in sulfuric acid copper (II) solution with polarization using industrial alternating current was shown for the first time. In this case, in each cathode half-cycle of alternating current, copper ions can be restored on the surface of the titanium

electrode polarized at high current densities, and as a result copper powders are formed. At that moment, when a copper electrode with a larger area is in the anodic half-period, copper (II) ions dissolve. When the titanium electrode is under alternating current in the anode half-period, its surface is a film of titanium oxide ( $Ti_xO_y$ ), which has semiconductor properties, which prevents the passage of electric current in this direction. These processes are periodically repeated on the surface of the electrodes with a frequency of 50 Hz.

**Experiments and process mechanism.** In the proposed research work, many scientific ideas were filtered out and a special facility was assembled for the first time. The possibility of obtaining copper powder in its cathode and in the anodic half-periods during the polarization of industrial alternating current using this device is considered. A schematic diagram of this setup is shown in figure 1 below.

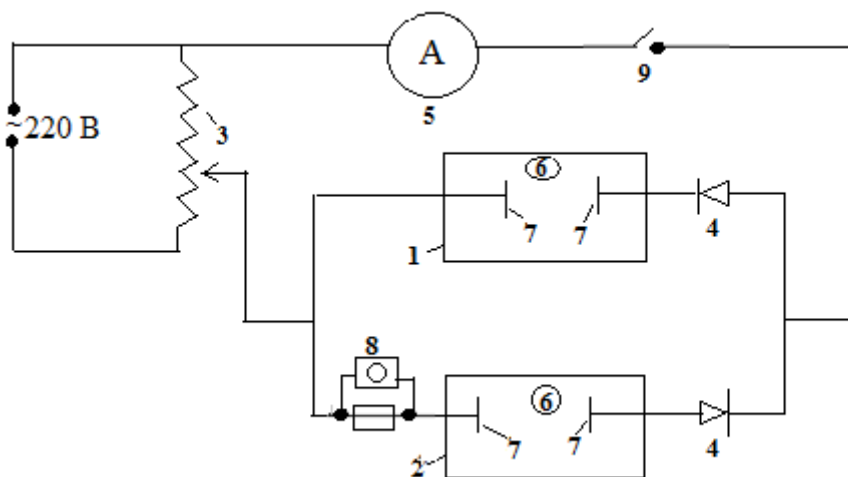


Figure 1 – Schematic circuit diagram of the installation for obtaining copper powder in its two periods by polarization of alternating current: 1 – first electrolyzer, 2 – second electrolyzer, 3 – laboratory transformer, 4 – diodes, 5 – amperometer, 6 – electrolyte, 7 – copper electrodes, 8 – oscillograph, 9 – key

This installation is a combination of two electrolyzers parallel to each other (1,2) from laboratory transformer (3), which allows regulating the alternating voltage (power) of two diodes (4) of the brand KD 213 A, placed in the opposite direction from each the other in the electrical circuit, and an ammeter (5) indicating the amount of current passing through the circuit. Sulfuric copper (II) electrolyte (6) is poured into each electrolyzer and two copper electrodes (7) are installed.

A known amount of alternating current, issued by the laboratory transformer, is divided equally into two electrolyzers, which are installed parallel to each other. In general, the alternating current passing through an electrochemical circuit is transmitted 50 times per second in one direction and the same amount, therefore, in the opposite direction. Since the diodes connected to the circuit are connected to each other, the alternating current passes through the first electrolyzer in its half-period, and through the second electrolyzer in its half period.

An oscillogram of currents passing through two electrolyzers was recorded during the polarization of alternating currents. The oscillogram of currents passing through each electrolysis circuit shows the passage of a pulsed current using electrodes (figure 2). Therefore, the electrodes are polarized in turn by anodic and cathodic pulsed currents.

As shown in Figure 1, if in the first electrolyzer on the right side in one half period the copper electrode serves as a cathode, then in the second half period in the second electrolyzer the copper electrode located on the left side is considered to be a cathode, as a result copper reduction proceeds on them:



Since the current density in the cathodes is higher than the limiting current density, copper (II) ions are oxidized to form copper powders.

The electrolysis was carried out using the above installation. The main goal is to obtain copper powders in cathodic and anodic half periods by polarization with alternating current.





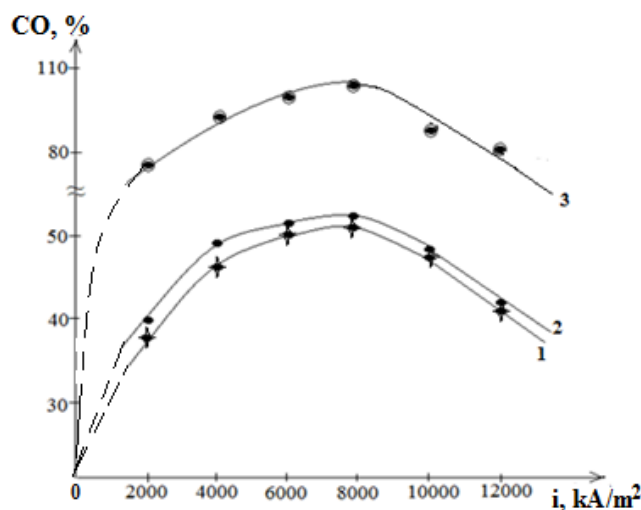
$$i = 600 \text{ A/m}^2; \text{H}_2\text{SO}_4 - 100 \text{ g/l}; \text{Cu (II)} - 15 \text{ g/l}; t = 25 \text{ }^\circ\text{C}$$

Figure 2 – Oscillogram of the amplitude of the current passing through each circuit of the electrolyzer when polarized with alternating current

Two electrolysis are filled with copper (II) solution with dilute sulfuric acid, their concentration:  $\text{H}_2\text{SO}_4 - 100 \text{ g/l}$ ,  $\text{Cu (II)} - 15 \text{ g/l}$ . Copper electrodes area are the same ( $13.68 \text{ cm}^2$ ). Our preliminary studies have shown that copper powders are formed in both electrolyzers. In our first study, we investigated the effect of current density ( $2000\text{--}12000 \text{ A/m}^2$ ) on the formation of copper powder on a copper electrode. The current output during the formation of copper powders in each electrolyzer was calculated for the cathode half-period with alternating current.

As a rule, copper powder should be formed of the same size in both electrolyzers, however, due to errors in the distances between the electrodes in each cell and the inability to reproduce the ideal electrode areas, the experimental results show that small deviations are observed.

**Experimental procedure.** The research results showed that while current density increasing on the electrodes to a value of  $8000 \text{ A/m}^2$ , the current efficiency during copper powder formation increases, but a subsequent increase in the values leads to a decline (figure 3). The decrease in current efficiency during copper powders formation at high densities can be explained by the reaction of additional evolution of gaseous hydrogen. For example, when electrodes current density is  $2000 \text{ A/m}^2$ , the current efficiency of copper powders formed in the first electrolyzer is 38.0%, in the second – 40.1%, and thus the amount of metal powders deposited in two electrolyzers can be considered about the same, as it should be. While the current density in both electrolyzers was  $8000 \text{ A/m}^2$ , the current efficiency of the powders in the electrolyzer was 50.1 and 52.3%, respectively.



$$\text{H}_2\text{SO}_4 - 100 \text{ g/l}; \text{Cu (II)} - 15 \text{ g/l}; \tau = 30 \text{ min}; t = 25 \text{ }^\circ\text{C}$$

Figure 3 – The effect of current density on copper electrodes polarized with alternating current on the current output during copper powders formation: 1 – first electrolyzer; 2 – second electrolyzer; 3 – in two electrolyzers

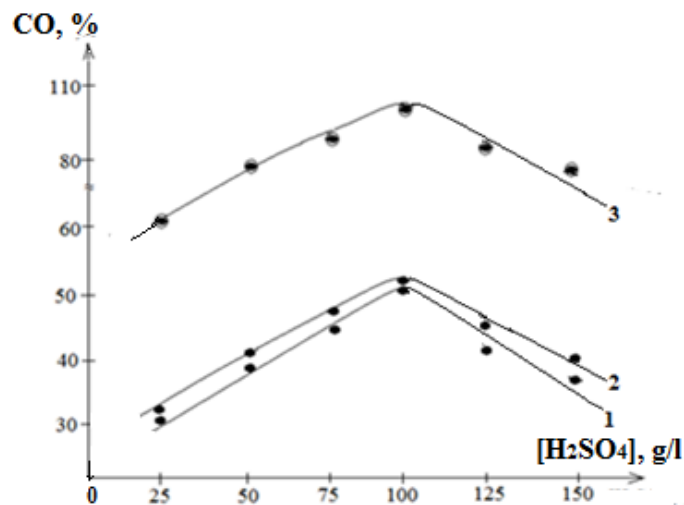


If you pay attention, the total current efficiency of copper powders formed in two electrolyzers exceeds 100%. This phenomenon can be explained by the reaction of chemical disproportionation:



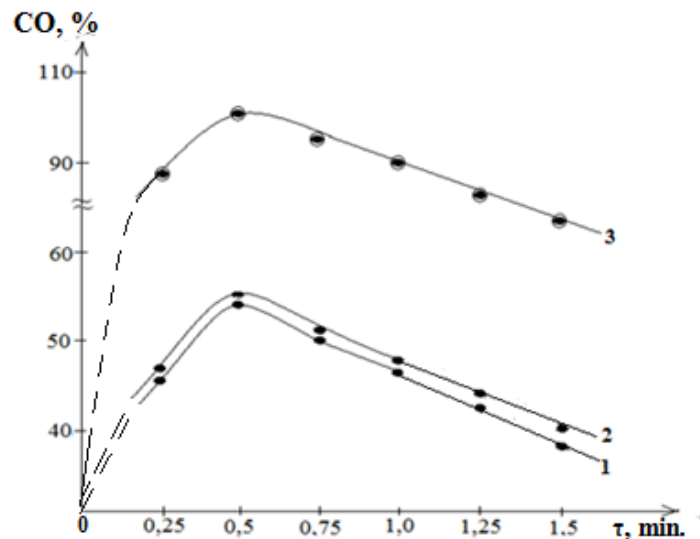
Our previous studies [18] showed that the potential fluctuation of copper electrode leads to the formation of copper powders based on reaction 2.

At the proposed facility, we investigated the effect of sulfuric acid concentration in the electrolyte within the range of 25–200 g/l on copper powders formation. When increasing the amount of sulfuric acid in the electrolyte to 100 g/l, the current efficiency of the formed copper powders increases slightly, and then this indicator decreases (figure 4). Current efficiency increasing can be explained by an increase in the conductivity of the electrolyte current, and a decrease due to the re-dissolution of the precipitated copper powders and a decrease in the high voltage of hydrogen ions, which can be explained by an increase in the release of gas.



$i = 8000 \text{ A/m}^2$ ,  $[\text{Cu}^{2+}] = 15 \text{ g/l}$ ;  $\tau = 30 \text{ min}$ ;  $t = 25 \text{ }^\circ\text{C}$

Figure 4 – The influence of sulfuric acid concentration on current output of copper powders formation during polarization with alternating current: 1 – first electrolyzer; 2 – second electrolyzer; 3 – in two electrolyzers



$i_{Ti} = 8000 \text{ A/m}^2$ ;  $i_{Cu} = 150 \text{ A/m}^2$ ;  $\text{H}_2\text{SO}_4 = 100 \text{ g/l}$ ;  $[\text{Cu}^{2+}] = 15 \text{ g/l}$ ;  $t = 25 \text{ }^\circ\text{C}$

Figure 5 – The influence of electrolysis duration on current output of copper powders formation during polarization with alternating current: 1 – first electrolyzer; 2 – second electrolyzer; 3 – in two electrolyzers

It should be noted when sulfuric acid concentration in the solution was 100 g/l, the current efficiency in the first electrolysis was 49.1%, in the second – 52.3%, while the total value of copper powders current efficiency in both electrolyzers amounted to 100.4%.

The effect of electrolysis duration on copper powders formation was investigated. The results of the study showed that copper powders deposition up to 0.5 hours increased the current efficiency, however, a further increase leads to deterioration (figure 5). Current efficiency decreasing is associated with a decrease in the concentration of copper (II) ions in the electrolyte and gaseous hydrogen proportion increasing.

**Conclusions.** To determine the shape and size of copper powders formed during electrolysis, photographs were taken of them on the latest version of a JSM-6610 LV scanning electron microscope (figure 6). During the course of electrolysis and a current density of 4000 A/m<sup>2</sup>, metal powders with a circular shape of 1–1.5 μm were found, while at higher densities, the formation of grouped together ultrafine copper powders, such as dendrites of ~ 1 μm size, was found.

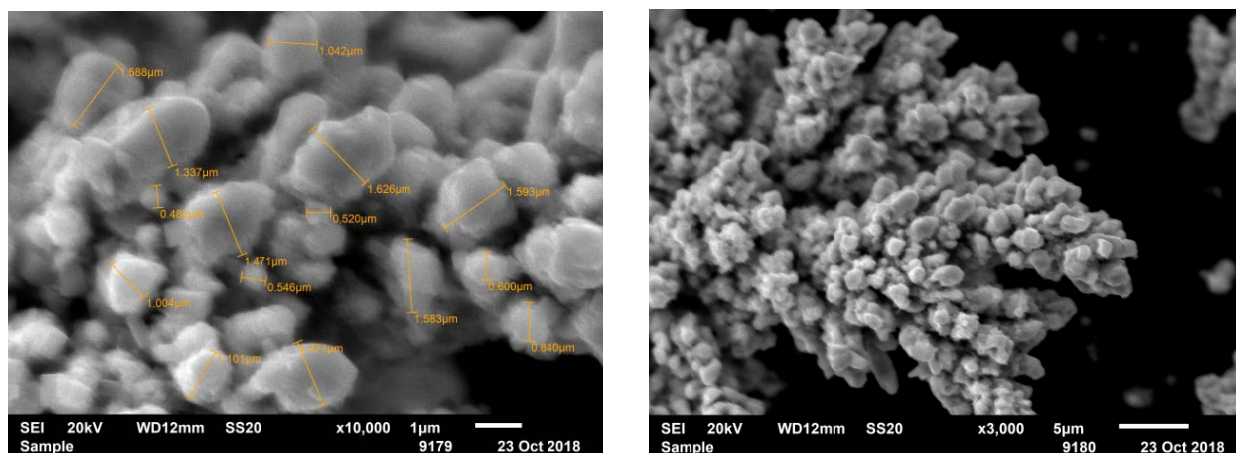


Figure 6 – Micrographs of electron microscopy of copper powder precipitated during electrolysis

Thus, for the first time, it was shown that it is possible to obtain copper powders with a high current output on the anodic and cathodic half-periods at an industrial alternating current with a frequency of 50 Hz on a special device recommended by us. In this case, copper powders formation with a dispersed particle size in the region of 1–1.5 μm was established.

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### ӨНДІРІСТІК АЙНЫМАЛЫ ТОКТЫҢ АНОДТЫҚ ЖӘНЕ КАТОДТЫҚ ЖАРТЫЛАЙ ПЕРИОДТАРЫНДА МЫС ҰНТАҚТАРЫНЫҢ ТҮЗІЛУІ

**Аннотация.** Алғаш рет мыс ұнтақтарын жиілігі 50 Гц-ке тең айнымалы токтың анодтық және катодтық жартылай периодтарында алуға арналған қондырғы жасалды. Қондырғы бір-бірімен параллель түрде жалғанған екі электролизерден құралған, әрбір электролизерде екі-екіден мыс электродтары орналастырылған. Сонымен қатар электрохимиялық тізбекке екі диод қосылған, олар токтың қарама-қарсы бағытта жүруін қамтамасыз етеді. Әрбір электролизерде түзілетін мыс ұнтақтарының ток бойынша шығымына ток тығыздығының, күкірт қышқылының және мыс иондарының концентрациясының әсері зерттелінді. Ток бойынша шығым ток тығыздығының 2000–8000 А/м<sup>2</sup> аралығында артатыны және бұл параметрдің ары қарай жоғарылауында азаятыны көрсетілді. Күкірт қышқылының концентрациясы 100 г/л-ге және мыс иондарының

концентрациясы 15 г/л құрағанда, электролизерлерде түзілетін ұнтақтың максималды ток бойынша шығымы 50,1–53,3%, ал жалпы ток бойынша шығым 100%-тен асады.

JSM-661LV маркалы сканирлеуші электрондық микроскоптың көмегімен алынған ұнтақтардың формалары және размерлері анықталды. Жүргізілген зерттеулер және түсірілген осциллограммалар негізінде айнымалы токтың анодтық және катодтық жартылай периодтарында мыс ұнтақтарының түзілу механизмі анықталды. Алғаш рет ұсынылған кондырғыда мыс ұнтағын айнымалы токтың екі жартылай периодтарында алу мүмкіндігі көрсетілді. Бұл кезде бөлшек размерлері 1,0–1,5 мкм құрайтын металл ұнтақтарының түзілетіні анықталды.

**Түйін сөздер:** айнымалы ток, мыс ұнтақтары, титан, диод, электрод, электролит, анодтық және катодтық жартылай периодтар, осциллограмма.

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### **ФОРМИРОВАНИЕ ПОРОШКОВ МЕДИ В АНОДНОМ И КАТОДНОМ ПОЛУПЕРИОДАХ ПРОМЫШЛЕННОГО ПЕРЕМЕННОГО ТОКА**

**Аннотация.** Впервые создана установка для получения порошков меди в анодном и катодном полупериодах промышленного переменного тока с частотой 50 Гц. Установка состоит из двух электролизеров, соединенных между собой параллельно, в каждом электролизере расположены по два медных электрода. В электрохимическую цепь также включены два диода, обеспечивающие протекание тока в противоположных направлениях. Исследовано влияние плотности тока на электродах, концентрации серной кислоты, ионов меди, на выход по току образования порошка меди в каждом электролизере. Установлено, что выход по току увеличивается в пределах плотности тока от 2000–8000 А/м<sup>2</sup> и уменьшается при дальнейшем повышении данного параметра. При концентрации серной кислоты, равной 100 г/л и концентрации ионов меди 15 г/л, максимальный выход по току образования порошка в электролизерах составляет 50,1–53,3%, а общий выход по току превышает 100%.

С помощью сканирующего электронного микроскопа марки JSM-661LV исследованы форма и размеры полученных порошков. На основании проведенных исследований и снятых осциллограмм установлен механизм образования порошков меди в анодном и катодном полупериодах переменного тока. Впервые показано, что предложенная установка позволяет получить порошки меди в двух полупериодах переменного тока. Установлено, что при этом формируются ультрадисперсные порошки металла с размерами частиц 1,0–1,5 мкм.

**Ключевые слова:** переменный ток, порошки меди, титан, диод, электрод, электролит, анодный и катодный полупериоды, осциллограмма.

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