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СЕРИЯ ХИМИИ И ТЕХНОЛОГИИ

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каб. 310, тел. 291-62-80, факс 291-57-22, e-mail:orgcat@nursat.kz
Адрес типографии: ИП «Аруна», г. Алматы, ул. Муратбаева, 75

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A. Bayeshov [1], T.E. Gaipov [1], A.K. Bayeshova [2], A.V. Kolesnikov [3]

[1]Institute of Fuel, Catalysis and Electrochemistry named after DV Sokolsky, Almaty, Kazakhstan;
[2]Kazakh National University named after al-Farabi, Almaty, Kazakhstan;
[3]D.Mendeleyev University of Chemical Technology of Russia, Moscow, Russia
bayeshov@mail.ru, tulkinjon.gaipov@gmail.com, azhar_b@bk.ru, artkoles@list.ru

SYNTHESIS OF NANO- AND ULTRADISPERSE COPPER POWDERS BY CEMENTATION OF COPPER (II) IONS BY THREE-VALENT TITANIUM IONS

Abstract. The process of cementation of copper (II) ions by tri-valent titanium ions is considered. The consistent patterns of the formation of nano- and ultra-disperse copper powders as a result of the interaction of copper (II) ions with titanium (III) ions have been established. It was demonstrated that during the reaction atomic copper is formed, the particles of which are combined with the formation of fine aggregates of certain sizes, which are stabilized in the form of spheres.

In the course of the research, the possibility of obtaining titanium (III) sulfate, which is necessary for carrying out the cementation of copper (II) ions, is shown in an inexpensive, simplified way and the results of studying the influence of various parameters on this process are presented. The possibility of regeneration of tetra-valent titanium ions formed as a result of the reaction using an electrolyzer equipped with an anion exchange membrane is demonstrated.

The effect of the initial concentration of copper ions (II) and titanium ions (III) on the formation of copper powder was studied. The shapes and sizes of the obtained copper powders were determined using an electron microscope. The constant of the reversible oxidation-reduction reaction was calculated and it was established that copper (II) ions contained in the solution are almost completely formed as nano-scale copper powders.

A schematic diagram of the technology for the production of fine copper powders, corresponding to the requirements of modernity, is proposed.

Key words: titanium ions, copper, powders, cementation, electrolysis, alternating current, electrolyte, reduction.

Introduction. The formation of metal powder is one of the spheres of metallurgical engineering. Metallic powder is widely used in mechanical engineering, and metallurgical engineering chemistry. Metal powder also has its place in the field of metallurgical engineering [1-9].

If in a nutshell we turn our attention on the information on the use of metal powder: it is widely used in the manufacture of complex shapes parts in mechanical engineering. It turns out that it is possible to easily manufacture parts of very complex shapes by placing the metal powder in a certain shape and exposing high pressure at high temperatures. With this heat treatment, the temperature of the medium must be below the melting point of the metal produced. It is known that obtaining of complex shapes parts by planing and cutting hard metal costs more than 100 times or more in comparison with powder technology [6].

Along with powder metallurgy, flat dispersed metal powders are used as catalysts in chemical production (iron, nickel, copper, etc.), in oxygen-flux welding, magnetic defectoscopy (iron), in the manufacture of products from polymer materials, in the production of lacquers (zinc, lead, iron, nickel), in the production of batteries (lead), in the production of pyrophores. Powders of flat disperse iron, copper, nickel increase the mechanical strength of products when forming them from plastic, rubber, nylon. If irons, zinc, bismuth powders, are added to the rubber glue, the quality of rubber products will improve. In hydrometallurgy, zinc powder is used in the production of zinc, in the separation from solutions

containing copper and cadmium ions, by cementation; however such plants are widely used to separate gold from cyanide solutions [8-11].

If we talk about copper powder, in the ancient period copper powder was widely used as decorative cosmetics. It is impossible to imagine ceramics and fine arts without copper powder. Today, the use of copper powder becomes wider with every passing day.

Copper powders in an active form are widely used by powder metallurgy in the manufacture of mechanisms and for other purposes, mainly in electrical engineering, instrument making, mechanical engineering and aviation production, chemical manufacturing, nanotechnology. Also used in the manufacture of anti-wear agents, in the manufacture of automobile tires and many other areas [1-3, 16].

Recently, copper powders in the form of ultra-disperse spheres are used in the implementing of 3D technology, in reducing the friction force and wear of machine parts [16].

Copper powders can be obtained by electrochemistry and cementation. It should be noted that more than 90% of the copper powder currently produce is obtained only by the electrochemistry methods. In laboratory environment, copper powder is obtained by cementation of its ions with zinc powder. The standard copper potential is $E^0_{Cu/Cu^{2+}} = 0.34V$, and the zinc potential is $E^0_{Zn/Zn^{2+}} = -0.76V$. Since the potential difference value ($\Delta E = 0.34 - (-0.76) = 1.2V$) is very large, the size of the formed copper powder is 30-50 microns [17]. In the proposed article, we examined the process of cementation of copper (II) ions with tri-valent titanium ions. Preliminary studies have shown the formation of very flat, ultra-disperse copper powders. If comes to think of it, obtaining copper powder by cementation with titanium ions (III) has a promising future. Because copper powders in the form of a dispersed sphere are in great demand. But unfortunately, sulphate compounds of tri-valent titanium ions are not produced in a great volume and the cost of such compounds is very high. For this reason, we, in this proposed work, together with the study of the process of cementation of copper ions (II) with titanium ions (III), also considered the possibility of obtaining titanium sulfate (III) by simple affordable means.

Procedure of work execution. The study of the formation of copper powders by cementing copper ions (II) with tri-valent titanium ions was carried out in a 200 ml beaker. During the reaction, the solution was stirred with a magnetic stirrer. To carry out laboratory work, we used "chemically pure" copper sulfate ($CuSO_4 \cdot 5H_2O$). The sulphate of trivalent titanium required for investigation was obtained by polarization of two titanium electrodes by a 50-Hz alternating current in a solution of sulfuric acid. In this paper, the regularity of the formation of titanium sulphate (III) is examined, the effect of various electrochemical processes on it is studied, and their results are presented. The main investigations were carried out under atmospheric conditions at room temperature. The purity of titanium electrodes used to obtain titanium sulfate (III): Ti – 97.65%.

The effect of the initial concentration of copper (II) ions and titanium (III) ions on the formation of copper powder was investigated. The shapes and sizes of the obtained copper powders were evaluated through an electron microscope.

Theoretical. When the solution is combined with copper ions and the ions of trivalent titanium, the following reaction is obtained:

$$Cu^{2+} + 2Ti^{3+} \rightleftarrows Cu^0 + 2Ti^{4+} \quad (1)$$

According to the literature $Cu^{2+} \rightleftarrows Cu^0$, $Ti^{4+} \rightleftarrows Ti^{3+}$ the potential values of corrosion-recovery electrode systems [17]: $E^0_{Cu^{2+}/Cu} = 0{,}34V$, $E^0_{Ti^{4+}/Ti^{3+}} = 0{,}04V$.

In order to calculate the constant (K) of the above (1) recurrent redox reaction (red-ox), we determined lgK by the following formula [18]:

$$\lg K_{red\text{-}ox} = \frac{n\,(E_1^0 - E_2^0)}{0{,}059} \quad (2)$$

Further, the value of K (n=2) is set for the reaction (1):

$$K = 10^{\frac{(E_1^0 - E_2^0)n}{0{,}059}} \quad (3)$$

(2) by the equation:

$$\lg K_{Cu^{2+}/Ti^{3+}} = \frac{2(0{,}34 - 0{,}04)}{0{,}059} = 10{,}2$$

As a result, the value of the «red-ox» reaction constant (1):

$K \simeq 10^{10}$

This value of «K» proves that reaction (1) moves completely from the left to the right and that this is a complete, irreversible reaction. Consequently, this shows that repeated interaction of the formed copper powders with four-valence titanium ions, and a new formation of copper (II) and titanium (III) ions are impossible. In other words, reaction (1) will be equal only when the product of $[Cu^0] \cdot [Ti^{3+}]$ ions is larger by 10^{10} products of $[Cu^{2+}] \cdot [Ti^{3+}]$ ions.

In previous studies [15, 20-22], we demonstrated the formation of ultra-disperse copper powders on the cathode-anode area, and in the size of the electrolyte. And in this article, we propose the regularities of the formation of copper powders by cementing copper ions (II) with ions (III) titanium.

Experimental. The solution containing copper (II) ions was poured into a beaker, stirred and gradually added to a solution with tri-valent titanium sulfate. The solution of titanium sulphate (III) was added to the decoloration of copper (II) sulphate of blue color. At that time an interesting phenomenon was observed. As indicated above, a solution of copper (II) sulfate is blue, and a solution of sulphate of tri-valent titanium is violet. When the two solutions are combined, the electrolyte is discolored. It is known that a solution of four-valent titanium is colorless. When the concentration of copper ions in the solution is 1 g/l, within two to three minutes the formation of copper powders is not visible by eyes. Only after four to five minutes, you can observe the formation of copper powders of colloidal light yellow color throughout the electrolyte. Consequently, copper (II) ions are reduced to very dispersed metal powders and a colloidal copper solution is formed.

At a certain point in the electrolyte the formation of copper powders becomes not visible by eyes, it can be assumed that this is due to the formation of atomic copper. Only after a certain time, the copper atoms begin to connect with each other, and a light yellow-colored colloidal metal solution is formed. After a couple of hours, these copper particles are even more connected and precipitated. We believe that the dimensions observed through the microscope are not their exact dimensions, but only their combined forms.

The table below shows the values of copper powders formed by combining the solution with different copper (II) ions and titanium (III) ions.

Table 1 - The impact of the initial concentration of copper (II) ions on the formed copper powders: V = 100 ml

Concentration of copper (II) ions in solution, g / l	The value of copper in solution, g	Mass of the formed copper powder, g
1,0	0,1	0,098
2,5	0,25	0,225
5,0	0,5	0,480
7,5	0,75	0,710
10	1,0	0,930
20	2,0	1,920

The results of the study showed that during the reaction, the copper (II) ions completely transform into the form of nano-sized copper powders.

Copper powders obtained on the basis of cementation, filtered, rinsed, dried in an inert medium (box).

In figures 1-4, microphotographs of copper powders formed at different initial concentrations (1 g/l, 5 g/l, 10 g/l) of copper (II) ions in solution are presented.

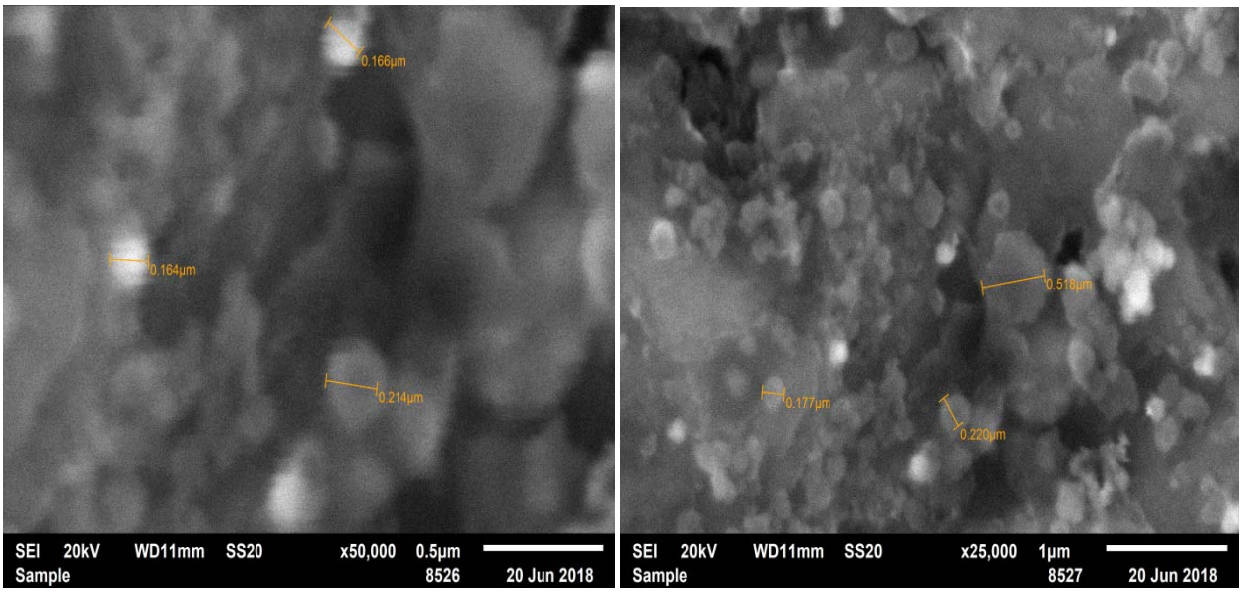

Figure 1 - Microphotograph of copper powders with the initial concentration of copper (II) ions in a solution of 1 g/l: the average size of copper powders is 0.1 - 0.4 µm.

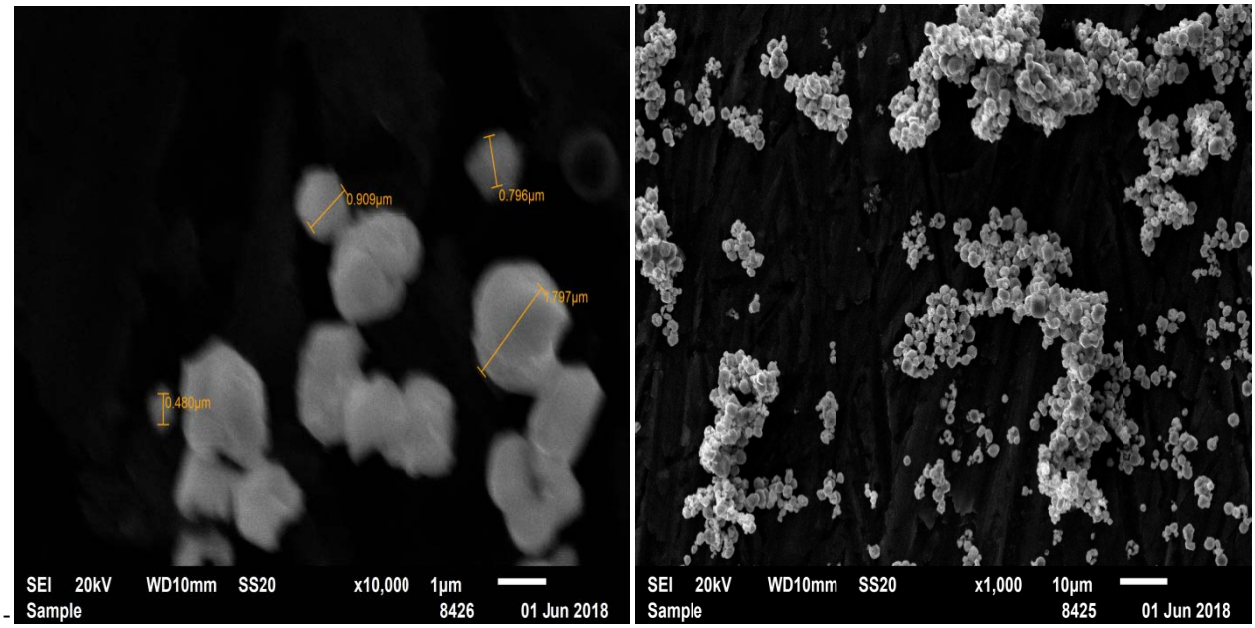

Figure 2. Microphotograph of copper powders with the initial concentration of copper (II) ions in a solution of 5 g/l: the average size of copper powders is 0.4-0.9 µm.

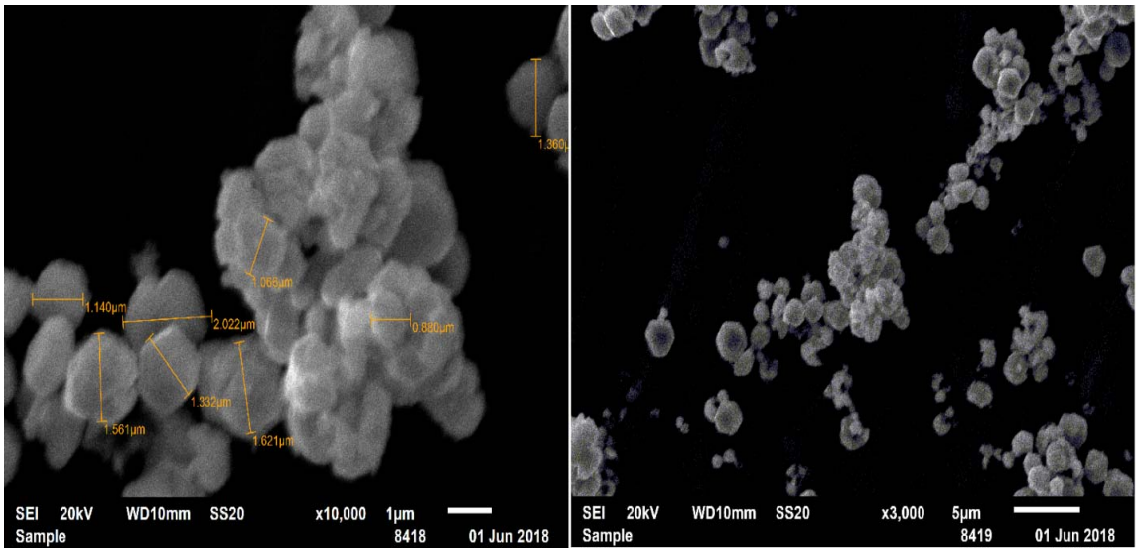

Figure 3. Microphotograph of copper powders with the initial concentration of copper (II) ions in a solution of 10 g / l: the average size of copper powders is 0.8 - 2 µm.

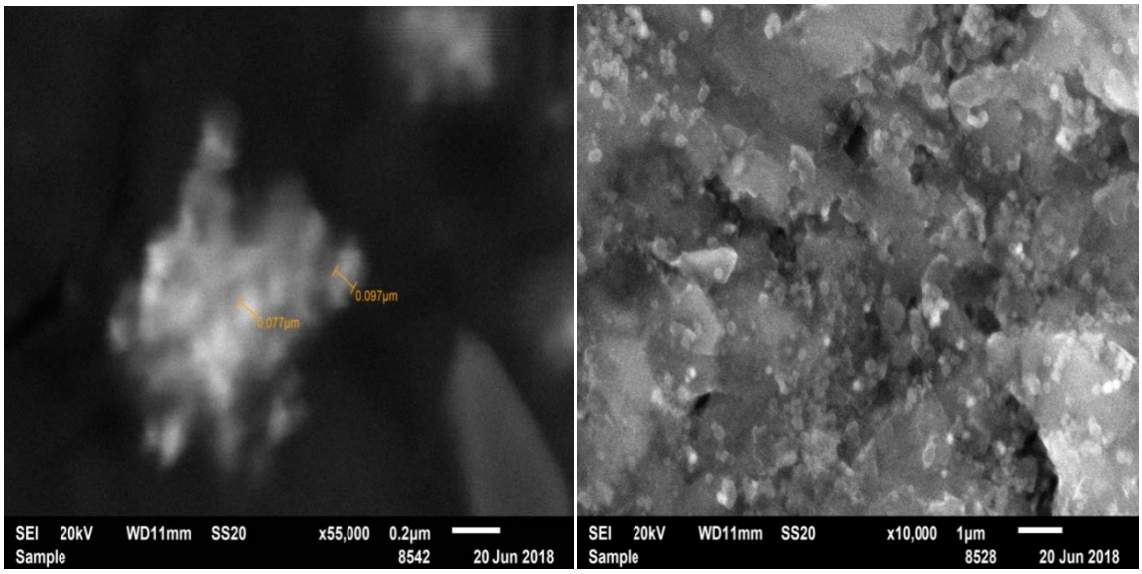

Figure 4 - Microphotography of copper powders with the initial concentration of copper (II) ions in a solution of 1 g/l: the average size of copper powders is 0.077-0.097 µm.

According to the results of the electron microscope examination, it was observed that with an increase in the initial concentration of copper (II) ions, the sizes of copper powders increased from 0.1 µm to 2 µm. In general, this is not the formation of copper powders in a large volume with a high initial concentration of copper (II) ions, we assume, that with an increase in the initial concentration of copper (II) ions it depends on the increase in the rate of the process of aggregation of copper atoms with each other. Because the sequential results of the study showed that copper powders have nanoscale form in a colloidal solution (Figure 4).

In order to make full use of the aforementioned method for producing a copper powder, it is necessary to create a simple method for preparing a tri-valent sulfate compound. The compounds of tri-valent ions are not constant, they are easily and rapidly oxidized to tetra-valent states. Therefore, this connection must be used immediately after receipt.

The influence of various parameters in the process of obtaining titanium (III) sulphate by the alternating current polarization of titanium electrodes in a solution of sulfuric acid is studied below.

The Figure 5 shows the effect of the current density in the electrodes on the current consumption of the dissolution of the formed $Ti_2(SO_4)_3$ compound in a 300 g/l sulfuric acid solution of the titanium electrode. When the current density is polarized with an alternating current of up to 200 A/m^2, dissolution of the titanium electrodes is not observed. After that, at 600 A/m^2 the dissolution rate by the current of titanium electrodes increases to 30%, and up to 1000 A/m^2 reaches the same value.

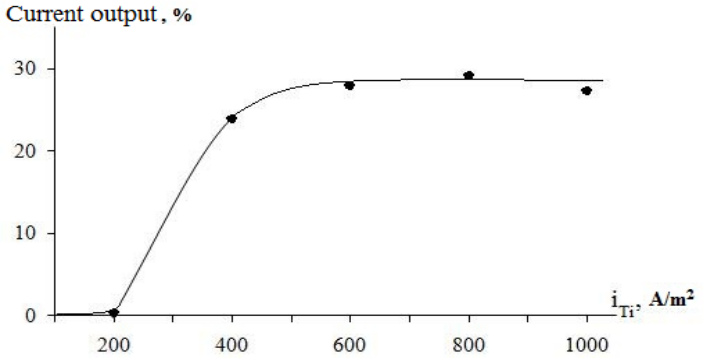

Figure 5 - The effect of the current density in the electrodes on the current consumption of the dissolution of the formed titanium (III) sulfate compound in a solution of sulfuric acid of titanium electrodes polarized by the industrial alternating current: H_2SO_4 = 300 g/l; τ = 0,5 hours; t = 25^{0}C.

Dissolution during the polarization of titanium electrodes by alternating current can be explained by the fact that in connection with the oxidation in the cathode half-period of the oxide film on the surface and anodic dissolution during the anode period. With prolonged electrolysis, it is possible to observe the precipitate of crystals of titanium (III) sulphate in the form of needles at the bottom of the electrolyzer.

The increase in the concentration of sulfuric acid in the solution increases the current consumption rate by the dissolution of titanium (Figure 6). This phenomenon can be explained by a decrease in the constancy of the titanium oxide layer on the surface of titanium electrodes due to the increase in the concentration of sulfuric acid.

We present the scheme of a new technology for obtaining ultra-disperse copper powder based on the above laboratory studies (Figure 7). Into electrolysis process, where two (2) titanium electrodes are located, a solution of sulfuric acid (3) is poured and alternating current in a certain volume is directed through the LAVD along the chain. At that time, tri-valent titanium sulfate is formed in the electrolysis. This electrolyte is directed to the reactor (4) with copper (II) sulfuric acid sulfate. In the reactor, ultra-disperse copper powders (1) are formed in reaction. After a certain time, precipitating dispersed copper powders are decanted, then filtered, washed, dried, as the result we have a very dispersed copper powder. The electrolyte released from the decantation, the electrode core, is sent to the cathode cavity of the electrolysis, separated by an anionite membrane, ions of tetra-valent titanium are oxidized-regenerated in the cathode of titanium to the state of tri-valence, and sent to the reactor (4) to obtain copper powders.

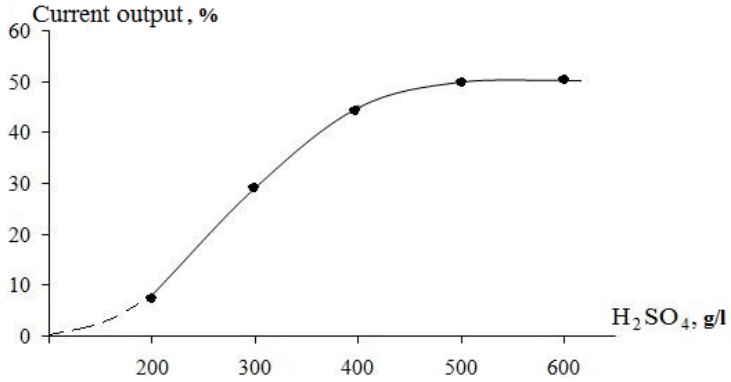

Figure 6 - The effect of the concentration of sulfuric acid in the solution on the dissolution of titanium electrodes: i_{Ti} = 800 A/m^2; τ = 0,5 hours; t = 25 ^{0}C.

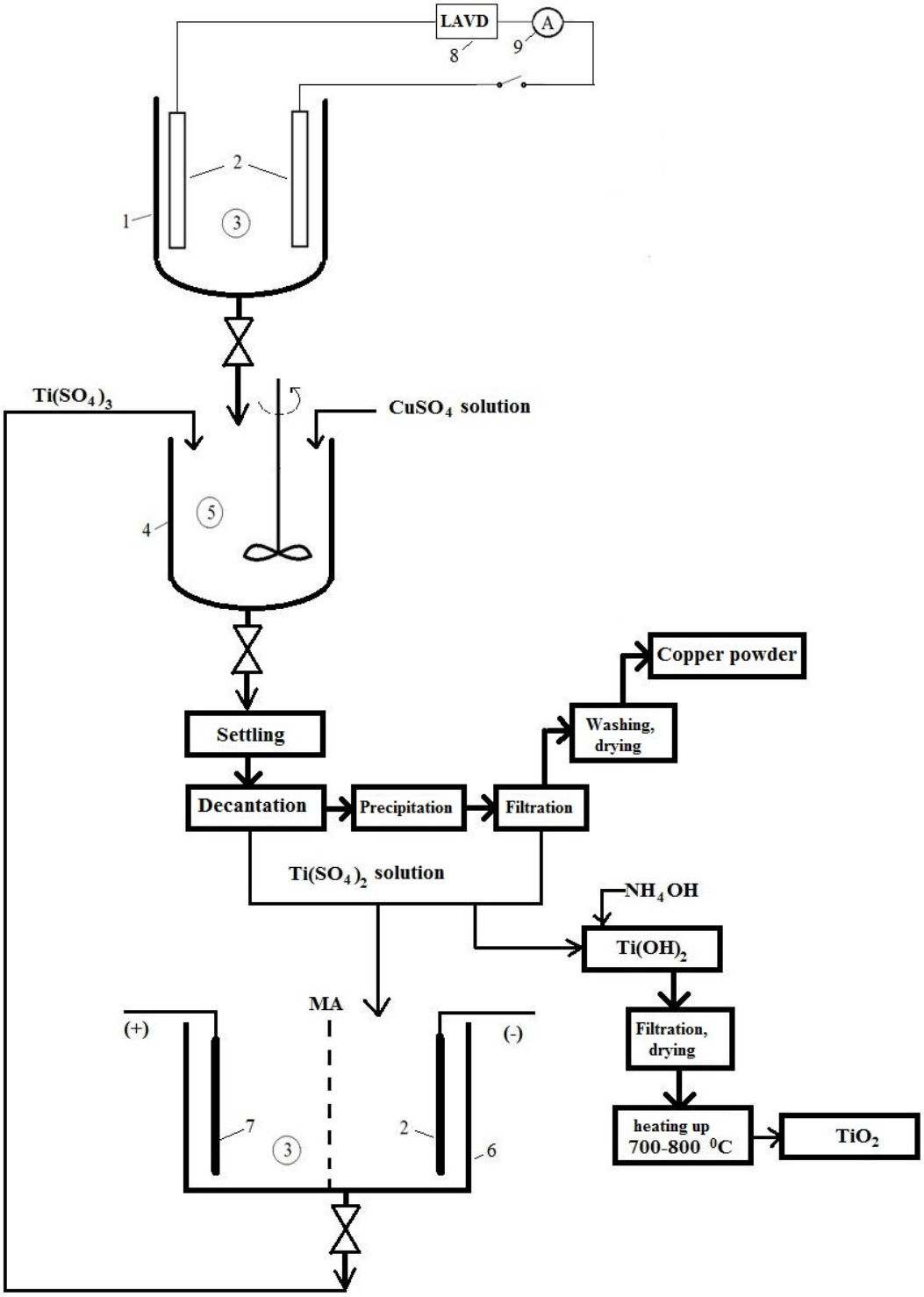

Figure 7 - Scheme of the principle technology of obtaining ultra-disperse copper powder by cementing a solution of copper (II) sulfate with titanium(III) ions: 1 - electrolyzer; 2 - titanium electrodes; 3 - solution of sulfuric acid; 4 - the reactor forming ultra-disperse copper powders; 5 - stirrer; 6 - electrolyzer designed to produce tri-valent titanium ions, separating the cores of the electrodes by an anionite membrane; 7 - lead electrode; 8 - LAVD; 9 - ampere-meter.

From the excess amount of tetra-valent titanium sulfate, as shown in the flow chart, by the addition of ammonia, it is possible to obtain the titanium (III) hydroxide, then the titanium dioxide (IV). It should be noted that the titanium (III) hydroxide is a very good sorbent, and its dioxide is the necessary pigment for obtaining a white color.

In conclusion, as a result of thorough comprehensive studies, the interaction of copper (II) and titanium (III) ions was studied, the patterns of formation of nano- and ultra-dispersed copper powders were established. During the reaction, the formation of atomic copper is established, their particles are connected to each other and flat dispersed aggregates are formed, stabilized in a certain volume in the form of a sphere. It has been shown that it is possible to obtain, in a simple and easy way, the tri-valent titanium compounds necessary for the preparation of the powder and to regenerate the tetra-valent titanium ions formed by the reaction using anionite membranes. A scheme of complex technology is presented in accordance with modern requirements for the preparation of dispersed copper powder.

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А. Баешов[1], Т.Э. Гаипов[1], А.К. Баешова [2], А.В. Колесников[3]

[1]Д.В.Сокольский атындағы Жанармай, катализ және электрохимия институты, Алматы, Қазақстан
[2]Әл-Фараби атындағы Қазақ ұлттық университеті, Алматы, Қазақстан
[3]Д.И. Менделеев атындағы Ресей химия-технологиялық университеті, Мәскеу, Ресей

МЫС (II) ИОНДАРЫН ҮШ ВАЛЕНТТІ ТИТАН ИОНДАРЫМЕН ЦЕМЕНТАЦИЯЛАУ АРҚЫЛЫ НАНО – ЖӘНЕ УЛЬТРАДИСПЕРСТІ МЫС ҰНТАҚТАРЫН АЛУ

Аннотация. Мыс (II) иондарын үш валентті титан иондарымен цементациялау процесі қарастырылды. Мыс (II) иондары мен титан (III) иондарының әсерлесуі зерттеліп, нано- және ультрадисперсті мыс ұнтақтарының түзілу заңдылықтары анықталды. Реакция кезінде атомарлы мыстың түзіліп, оның бөлшектері бір – бірімен белгілі өлшемдегі майда дисперсті агрегаттарға бірігіп, сфера формасында тұрақтанатыны айқындалды.

Зерттеу барысында мыс (II) иондарын цементациялауға қажетті титан (III) сульфатын қарапайым арзан жолмен алу мүмкіндігі қарастырылып, осы процеске әртүрлі параметрлердің әсерін зерттеу нәтижелері келтірілді. Реакция нәтижесінде түзілген титанның төрт валентті иондарын анионитті мембранасы бар электролизерді қолданып, регенерациялауға болатындығы көрсетілді.

Мыс ұнтағының түзілуіне мыс (II) иондарының және титан (III) иондарының бастапқы концентрацияларының әсері зерттелді. Алынған мыс ұнтақтарының формасы мен өлшемдері электрондық микроскоп арқылы сарапталды. Қайтымды тотығу-тотықсыздану реакциясының константасы есептеліп, ерітіндідегі мыс (II) иондары түгел дерлік наноразмерлі мыс ұнтақтары түріне өтетіндігі анықталды.

Өте дисперсті мыс ұнтағын алудың казіргі заманның талабына сай кешенді технологиясының принципиалды сызба-нұсқасы ұсынылды.

Түйін сөздер: титан иондары, мыс, ұнтақ, цементация, электролиз, айнымалы ток, электролит, тотықсыздану.

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А.Баешов[1], Т.Э.Гаипов[1], А.К.Баешова[2], А.В. Колесников[3]

[1]Институт топлива, электрохимии и катализа имени Д.В.Сокольского, Алматы, Казахстан;
[2]Казахский Национальный университет имени аль-Фараби, Алматы, Казахстан;
[3]Российский химико-технологический университет имени Д. И. Менделеева, Москва, Россия

ПОЛУЧЕНИЕ НАНО- И УЛЬТРАДИСПЕРСНЫХ ПОРОШКОВ МЕДИ ЦЕМЕНТАЦИЕЙ ИОНОВ МЕДИ (II) ИОНАМИ ТРЕХВАЛЕНТНОГО ТИТАНА

Аннотация. Рассмотрен процесс цементации ионов меди(II) ионами трехвалентного титана. Установлены закономерности формирования нано- и ультрадисперсных порошков меди в результате взаимодействия ионов меди (II) с ионами титана (III). Показано, что в процессе реакции образуется атомарная медь, частицы которой объединяются с формированием мелкодисперсных агрегатов определенных размеров, которые стабилизируются в виде сфер.

В процессе исследования показана возможность получения недорогостоящим, упрощенным способом сульфата титана (III), необходимого для проведения реакции цементации ионов меди (II) и приведены результаты изучения влияния различных параметров на данный процесс. Показана возможность регенерации четырехвалентных ионов титана, образующихся в результате реакции, с использованием электролизера, снабженного анионитовой мембраной.

Исследовано влияние исходной концентрации ионов меди (II) и ионов титана (III) на формирование порошка меди. Формы и размеры полученных порошков меди установлены с помощью электронного микроскопа. Рассчитана константа обратимой окислительно-восстановительной реакции и установлено, что ионы меди (II), содержащиеся в растворе, практически полностью формируются в виде наноразмерных порошков меди.

Предложена принципиальная схема технологии получения мелкодисперсных порошков меди, соответствующая требованиям современности.

Ключевые слова: ионы титана, медь, порошки, цементация, электролиз, переменный ток, электролит, восстановление.

Information about the authors:

Bayeshov A. - Chief Researcher, Doctor of Chemical Sciences, Laboratory of electrochemical technology, JSC "D.V. Sokolsky Institute of Fuel, Catalysis and Electrochemistry", Institute of Fuel, Electrochemistry and Catalysis named after DV Sokolsky, Almaty, Kazakhstan. Tel: 87017605635, e-mail: bayeshov@mail.ru, orcid: 0000-0003-0745-039X

Bayeshova A.K. - Doctor of Chemical Sciences, Al-Farabi Kazakh National University, Almaty, Kazakhstan. Tel: 87017605625, e-mail: azhar_b@bk.ru, orcid: 0000-0002-9076-8130

Gaipov T.E. - Senior Researcher, Candidate of Chemical Sciences, Laboratory of electrochemical technology, JSC "D.V. Sokolsky Institute of Fuel, Catalysis and Electrochemistry", Institute of Fuel, Electrochemistry and Catalysis named after DV Sokolsky, Almaty, Kazakhstan. Tel:87024962449, e-mail: tulkinjon.gaipov@gmail.com, orcid: 0000-0002-9723-3745

Kolesnikov A. - Candidate of technical Sciences, D.Mendeleev University of Chemical Technology of Russia, Moscow, Russia, Tel: 8 985 243 46 46, e-mail: artkoles@list.ru, orcid: 0000-0002-4586-6612

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