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KINETICS OF ELECTRODEPOSITION OF INDIUM ON SOLID ELECTRODES FROM CHLORIDE SOLUTIONS

Abstract. Using the methods of cyclic voltammetry and chronoamperometry, the electroreduction of indium on titanium, platinum and glassy carbon electrodes from perchlorate-containing chloride electrolytes has been studied. The quasi-reversible character of the process under investigation for all the above electrodes are established on the basis of the analysis of the difference in potentials of the peaks on the forward and reverse course of the sweep and the relationship between the current of the reduction peak and the rate of polarization. The overvoltage of indium release in the peak on Pt, GC, Ti electrodes was 158, 111, 64 mV, respectively. The constants of the rate of charge transfer and mass transfer of indium electrodeposition are calculated using the Delahay and Cottrell equations. A comparative analysis of the obtained constants showed that they had large values in the case of a titanium electrode and amounted to $1.06 \cdot 10^{-3}$ and $4.5 \cdot 10^{-4}$ cm/s, respectively. The results obtained indicate the preferred use of titanium in the electro-chemical purification of rough indium.

Key words: indium, electrodeposition, mass transfer, charge transfer, diffusion coefficient, quasi-reversibility, rate constant.

Introduction. Electrochemical methods of deep purification of metallic indium have great potentialities and are especially valuable with a low content of micro-impurities. One of the main methods for obtaining high-purity indium is electrochemical refining, the implementation of which requires a deep and detailed study of the processes of discharge-ionization of indium on solid electrodes. This research is devoted to the development of the technology of electrochemical refining of the rough indium of Kazakhstan, which is a by-product of the lead-zinc enterprise Kazzinc JSC [1]. Obtaining domestic high-purity indium is a pre-requisite for the development of electronic and semiconductor industry in the Republic of Kazakhstan.

The quality of cathodic precipitation and economic indicators in the process of electrorefining of metals depend on the composition of the electrolyte, substrate nature and electrolysis conditions. A number of publications [2-5] are devoted to the study of indium electrodeposition on various electrodes in chloride and perchlorate electrolytes. In previous works [6, 7] we studied the electroreduction of indium on solid electrodes in acid chloride electrolytes. As a result of these studies, the kinetic characteristics of indium reduction on platinum and titanium electrodes were determined and the nature of the limiting stage was established. In this paper, the effect of the nature of the electrode and the concentration of the In^{3+} salt in solution on the kinetics of indium electroreduction in perchloratecontaining chloride electrolytes by cyclic voltammetry and chronoamperometry is considered.

EXPERIMENTAL PART

Electroreduction of indium was investigated by the method of cyclic voltammetry, chronoamperometry in a thermostated three-electrode electrochemical cell, using the potentiostat-galvanostat Autolab PGSTAT 302N. Working electrodes were platinum and glass-carbon electrodes 0.3 cm in diameter produced by Metrohm, as well as a titanium electrode 0.25 cm in diameter (of brand Ti BT1-0). The auxiliary electrode was a platinum plate with an area of 12.96 cm², and the reference electrode was a silver chloride electrode. All experiments were carried out at a temperature of 25°C with an accuracy of ± 0.1 °C. Perchlorate-containing chloride electrolytes of composition: 0.025; 0.05; 0.1 M In(ClO₄) $_3 + 2.0$ M NaCl (pH = 1.5) were prepared from 1.0 M indium perchlorate solution on the background of 2.0 M sodium chloride solution. Solution $In(ClO_4)_3$ was obtained by the interaction of metal indium of brand In-2 (with a 99.98% base metal content) with 60% solution of perchloric acid produced by Sigma Aldrich (99.99%). The basic background electrolyte was a solution of sodium chloride, obtained from a triply recrystallized chemically pure salt (99.9%). The pH values of the solutions (1.5-1.6) were created by acidifying them with perchloric acid. Before each measurement, the platinum and glassy carbon electrodes were purified in a solution of concentrated nitric acid, and the titanium electrode was machined using a micron skin, after which they were thoroughly washed with bidistilled water. To evaluate the reproducibility of the results, each measurement was carried out at least three times.

RESULTS AND DISCUSSIONS

Cyclic Voltammetry. Cyclic voltammetry is a simple and convenient method for studying the kinetics and mechanism of oxidation-reduction processes. In the case of reversible processes, the values of the potentials of the oxidation and reduction peaks that characterize the nature of the electroactive substance are independent of the sweep speed and their difference $(E_{n(\kappa)} - E_{n(a)})$ is a constant value. For irreversible processes this difference is greater than for reversible ones and depends on the sweep speed. When studying the processes of discharge-ionization of indium, cyclic voltammograms were obtained on platinum (Pt), glassy carbon (GC) and titanium (Ti) electrodes from perchlorate-containing chloride solutions. Increase the sweep rate of the potential from 5 mV/s to 40 mV/s induces the displacement of the indium reduction peak ($E_{n(\kappa)}$) in the electrolyte of the following composition: 0.1 M In (ClO₄) $_3$ + 2.0 M NaCl (pH = 1.5) into the cathode region for Pt, GC and Ti electrodes at 80, 58 and 54 mV, respectively. This is due to a slow decrease in the surface concentration of the oxidized form of

indium $(C_{In^{3+}}^{s})$, leading to the maximum value of the concentration gradient of the reagent at a more negative potential [8]. As can be seen from the cyclic voltammograms of the discharge-ionization of indium at different polarization rates of the titanium electrode (figure 1), the beginning of the reduction reaction is shifted, which is apparently related to the features of the formation of the new phase.



Figure 1 – Cyclic voltammograms of indium at 25° C on a Ti electrode in a solution of 0,1 M In(ClO₄)₃ + 2,0 M NaCl at different polarization rates

To determine the nature of the observed effect associated with the overvoltage of the formation of a new phase, cyclic voltammograms of indium precipitation and dissolution on an indium electrode were obtained (figure 2). The absence of a delay in indium reduction on an indium electrode indicates a low value of the overvoltage of phase formation. The irreversibility of the process of indium reduction on an indium electrode is reflected in the stretching of the currentvoltage curves. In the absence of diffusion restrictions, the shape of the currentvoltage curve is affected only by the kinetic parameters, such as the rate constant of charge transfer and the charge transfer coefficient. The potential difference between the peaks in the forward and reverse sweep runs depends on the rate constant of the electronic transition and on the sweep rate of the potential. From the analysis of the presented cyclic polarization curves it can be concluded that at high sweep rates the magnitude of the difference between the potentials of the peaks becomes very large and the degree of irreversibility of electron transfer increases. Consequently, high values of the potential difference of the reduction and oxidation peaks are associated with limitations on the electron transport kinetics.



Figure 2 – Cyclic voltammograms of indium at 25° C on an indium electrode in a solution of 0.1 M 0,1 M In(ClO₄)₃ + 2,0 M NaCl at different polarization rates

For irreversible processes, the relationship between the reduction peak current and the sweep rate of the potential is described by P. Delahay equation [9]. Analysis of the above experimental results showed a linear dependence of the current density of the reduction peak of indium $(i_{n(k)})$ on the square root of the value of the potential sweep speed $(\sqrt{\nu})$ (figure 3), which does not pass through the origin of coordinate, which indicates the quasi-reversible nature of the process under study.



Figure 3 – Dependence of the current density of the indium recovery peak (i_{nux}) on the \sqrt{v} at 25^oC on different electrodes in a solution of 0,1 M In(ClO₄)₃ + 2,0 M NaCl

The rate of many electrochemical reactions is very often determined by the rate of charge transfer or mass transfer. The determination of the process regime can be carried out by comparing the orders of the rate constants for the transport of matter and charge. For this purpose, we determined the values of the rate constants of charge transfer and mass transfer of reduction of indium ions in solution on the investigated electrodes.

For irreversible processes, the limiting stage is charge transfer, whose velocity is determined by the rate constant (K^0) and the transport coefficient (α). The Nernst boundary conditions for an irreversible one-stage and many-electron reaction are expressed by the following equations [10]:

$$i = nFk_f(t)C_o(0,t), \tag{1}$$

where

$$k_{f}(t) = k^{0} \exp\{-\alpha \frac{nF}{RT}[E(t) - E^{0'}]\}.$$
(2)

Proceeding from these expressions (1), (2), the peak potential and current density (3) for irreversible reactions can be represented as follows [10]:

$$i_{n} = (2,99 \cdot 10^{5}) \alpha^{1/2} n C_{0} \cdot D_{0}^{1/2} v^{1/2};$$
(3)

$$E_{n} = E^{0'} - \frac{RT}{\alpha n F} [0,780 + \ln(\frac{D_{0}^{1/2}}{k^{0}}) + \ln(\frac{\alpha n F \upsilon}{RT})^{1/2}];$$
(4)

$$i_{n} = 0,227 \cdot nFC_{0}^{*}k^{0} \exp[-(\frac{\alpha nF}{RT}) \cdot (E_{n} - E^{0})], \qquad (5)$$

where E_n - is the peak potential (B), E^0 - is the formal electrode potential (B), i_n - is the cathode current density of the peak (A), k^0 - is the rate constant of the charge transfer stage (cm/s), α - is the transfer coefficient, D_0 - is the diffusion coefficient (cm²/c), C_0^* - is the concentration of ions in the volume of the solution (mol/cm³), C_0 - is the ion concentration in the near-electrode region (mol/cm₃), ν - is the potential sweep rate (V/s).

As can be seen from equation (4), the reduction peak potential depends on the sweep speed. After logarithm of equation (5) we obtain the following expression [11,12]:

$$\ln i_{n} = \ln(0,227 nFC_{0}^{*}k^{0}) - (\frac{\alpha nF}{RT}) \cdot (E_{n} - E^{0}).$$
(6)

From cyclic voltammograms for the investigated electrodes, varying the rate of the potential sweep and the concentration of indium ions in the solution, a dependence $\ln i_n$ on $(E_n - E^{0'})$ (figure 4) was obtained, which made it possible to calculate the rate constants of charge transfer during the reduction of indium (table 1).



Figure 4 – Dependence of the logarithm of the current density of the cathode indium- $ln(i_{nuk})$ on $E_{nuk} - E^{0T}$ at 25^oC at different electrodes in a solution of 0,1 M In(ClO₄)₃ + 2,0 M NaCl

$C_{ln^{3+}}^{*}$,	k ⁰ , ·10 ⁻⁴ cm/s		
mol/l	Pt	GC	Ti
0,025	3,1	4,1	4,4
0,050	4,2	6,3	6,9
0,100	6,9	7,7	11,6

Table 1 - The rate constants of charge transfer during the reduction of indium in 2 mol/L NaCl

As can be seen from Table 1, the charge transfer rate constant for indium deposition on a titanium electrode in a 0.1 mol/l indium salt solution is much higher than that for platinum and glassy carbon electrodes. This indicates a greater degree of reversibility of the process on the titanium electrode.

Figure 5 shows cyclic voltammograms of discharge-ionization of indium on electrodes of various nature. The obtained experimental data indicate that the difference in potentials of the cathode and anode indium peaks is 252, 235 and 154 mV for solutions with an indium salt concentration of 0.1 mol/l for Pt, GC and Ti electrodes, respectively. For reversible three-electrode processes, it should not exceed 19.7 mV. The maximum shift to the anode region is observed for the titanium electrode, which also indicates a greater degree of reversibility of the reduction of indium on this electrode.

Overvoltage of the cathodic deposition of indium depends on the nature of the electrode. From the cyclic voltammograms of the discharge-ionization of indium (figure 5), the values of the overvoltage of indium release in the peak at Pt, GC and Ti electrodes, which amounted to 158, 111, 64 mV, respectively, were



Figure 5 – Cyclic voltammograms of indium at 25° C at various electrodes in a solution of 0,1 M In(ClO₄)₃ + 2,0 M NaCl at a polarization rate of 5 mV/s

determined. This indicates a small value of the energy barrier of the cathodic reaction on the titanium electrode. The results obtained can be related to the values of the electron work function for various metals: 5.12-5.93 eV Pt; 5.0 eV GC; 4.33 eV Ti [13]. However, this is only one of the characteristics of the electrode material, which affects the electrode processes. It is necessary to take into account the peculiarities of the structure of the double electric layer and the processes of specific adsorption.

The displacement of the potential of the indium reduction peak in the anode region depends on the concentration of indium ions in the solution (figure 6). An

Figure 6 – Cyclic voltammograms of indium at 25^{0} C C on a Ti electrode in a 2.0 M NaCl solution with a different content of In(ClO₄)₃, at a polarization rate of 5 mV/s

increase in the concentration of indium from 0.025 to 0.1 M at a potential sweep rate of 5 mV/s causes a shift in the potential of the reduction peak of indium ions on Pt, GC and Ti electrodes by 15, 11, 48 mV, respectively. The use of electrolytes with a high concentration of the potential-determining ion leads to an increase in the potential difference between the reduction of indium and electronegative impurities and favors the purification of the base metal from them. Also, the use of titanium as a cathode in the refining of indium will permit electrolysis at relatively high current densities with a high current yield of metal.

Chronoamperometry. Chronoamperometry makes it possible to find the kinetic parameters of the mass transfer stage, such as the effective mass transfer constant and the diffusion coefficient of the potential-determining ion [14]. To determine diffusion coefficients and mass transfer constants, we obtained chronoamperograms of reduction of indium ions on Pt, GC, and Ti electrodes from perchlorate-containing chloride electrolytes by varying the concentration of indium salt (figure 7).

Figure 7 – Chronoamperograms of electrodeposition of indium at 25^{0} C at various electrodes in a solution of ,1 M In(ClO₄)₃ + 2,0 M NaCl at E = -0.9 V

For the completeness of the reaction during chronoamperometric measurements, it is necessary to supply an electrode with a sufficient jump in potential, which ensures a zero value of the concentration of the oxidized form of the substance on its surface. In our case, it is -0.9 V. In this case, the Cotrell equation, which expresses the dependence of the current density on time for nonstationary diffusion [8, 15], is applicable.

The diffusion coefficient of the electroactive substance is found from the slope of the straight line $i(t)=f(t^{1/2})$. Table 2 presents the results of an analysis of chronoamperograms of indium reduction on the investigated electrodes from chloride electrolytes containing various concentrations of indium perchlorate.

$C_{ln^{3+}}^{*}$,	$D_{In^{3+}}$, $\cdot 10^{-5}$ cm ² /s		
mol/L	Pt	GC	Ti
0.025	2.4	1.7	3.2
0.050	1.3	1.6	3.2
0.100	0.6	0.6	0.3

Table 2 – Values of diffusion coefficients of indium ions in 2 mol / 1 NaCl solution

The diffusion coefficient is determined by the properties of the medium and by the type of diffusing particles. The absence of significant differences in the values of the indium diffusion coefficients during its reduction at various electrodes indicates the validity of the application of the Cotrell equation.

From the equation of the limiting diffusion current for nonstationary diffusion, the effective mass-transfer rate constant for three electrodes was calculated (table 3).

Table 3 – The values of the rate constants of mass transfer during electroreduction of indium

Electrode	Pt	GC	Ti
$C_{In^{3+}}^*, \operatorname{mol/L}$	m_{ef} , $\cdot 10^{-4}$ cm/s	m_{ef} , $\cdot 10^{-4}$ cm/s	m_{ef} , $\cdot 10^{-4}$ cm/s
0.025	2.4	3.1	3.6
0.050	2.9	3.3	3.9
0.100	3.7	3.5	4.5

Comparison of the values of the constants of the transport of matter and charge testifies to the quasi-reversibility of the system under study. The constant of the mass transfer rate of indium at reduction on a titanium electrode is higher than that of platinum and glassy carbon electrodes, which indicates an accelerated transfer of matter on this electrode and explains the large limiting currents when indium is reduced on the surface of titanium. From the values obtained for the mass transfer constants of indium ions when it is discharged on substrates of various nature, it is clear that the use of titanium in indium refining is preferable.

Conclusion. Based on cyclic voltammograms, the difference in the potentials of the oxidation peak and indium reduction on platinum, glassy carbon and titanium electrodes, as well as a comparative analysis of the calculated charge transport and mass transfer constants, is established that the discharge of indium ions proceeds quasi-reversibly. The greatest degree of reversibility is observed for a titanium electrode.

The values of the overvoltage of indium emission in the peak, at Pt, GC, Ti electrodes can be arranged in descending order as follows: 158, 111, 64 mV. Small overvoltages and the maximum value of the limiting current of indium deposition on a titanium electrode is the rationale for its choice in the refining of indium. With an increase in the InCl₃ concentration, the overvoltage of indium

reduction on the titanium electrode decreases, which leads to an increase in the difference in the deposition potential between the parent metal and electronegative impurities, which will improve the purity of the cathodic precipitates.

The values of the rate constant of charge transfer and mass transfer, found from cyclic voltammograms and chronoamperograms, in the process of indium reduction on a titanium electrode at 0.1 mol/L concentration of the potential-determining ion were $1.2 \cdot 10^{-3}$ and $4.5 \cdot 10^{-4}$ cm/s, respectively. The values of the above constants on Pt and GC cathodes are somewhat lower, so the use the titanium cathode will make it possible to purify indium at high current densities with a high current output.

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Резюме

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ХЛОРИДТІ ЕРІТІНДІЛЕРДЕН ҚАТТЫ ЭЛЕКТРОД БЕТІНДЕ ИНДИЙДІҢ БОЛІНУ КИНЕТИКАСЫ

Құрамында перхлораты бар хлоридті ерітінділерден индийдің электртотықсыздануын циклдің вольтамперометрия және хроноамперометрия әдістерімен титан, платина және шыныкөміртекті электродтарда зерттелді. Тура және кері жолдағы потенциал пиктерінің айырмашылығы, поляризация жылдамдығы мен тотықсыздану пикі тоғымен арасындағы байланыс арқылы зерттеліп жатқан процесстің жоғарыдағы электродтарда квазиқайтымды екені анықталды. Рt, ШК және Тi элекродтарында индийдің бөліну пикі аса кернеуі 158, 111 және 64 мB екені анықталды. Делахей және Коттрел теңдеулері арқылы индийдің электр тогы натысинда бөлінуін заряд тасымалдау жылдамдық константасы мен масса тасымалдау жылдамдық константасы есептелді. Анықталған константалардың салыстырмалы анализі нәтижесінде титан электродында жоғары мәндер байқалды: 1,06·10⁻³ и 4,5·10⁻⁴ см/с сәйкесінше. Алынған нәтижелер қаралашты индийдің электрохимиялық тазалау кезінде титанды қолдануын ұтымды екеніне көз жеткізді.

Түйін сөздер: индий, электродтау, массалық трансфер, зарядты беру, диффузия коэффициенті, квази-реверсивтілік, жылдамдық константасы.

Резюме

Х. Авчукир, Б. Д. Буркитбаева, А. М. Аргимбаева, Г. С. Рахымбай

КИНЕТИКА ЭЛЕКТРООСАЖДЕНИЯ ИНДИЯ НА ТВЕРДЫХ ЭЛЕКТРОДАХ ИЗ ХЛОРИДНЫХ РАСТВОРОВ

Методами циклической вольтамперометрии и хроноамперометрии исследовано электровосстановление индия на титановом, платиновом и стеклоуглеродном электродах в перхлоратсодержащих хлоридных электролитах. Установлен квазиобратимый характер исследуемого процесса для всех вышеуказанных электродов на основании анализа разности потенциалов пиков на прямом и обратном ходе развертки и взаимосвязи тока пика восстановления от скорости поляризации. Перенапряжение выделения индия в пике на Pt, СУ и Ti электродах составило 158, 111 и 64 мB, соответственно. Рассчитаны константы скорости переноса заряда и массопереноса электроосаждения индия с использованием уравнений Делахея и Коттрела. Сравнительный анализ полученных констант показал, что они имели большие значения в случае титанового электрода и составили 1,06·10⁻³ и 4,5·10⁻⁴ см/с, соответственно. Полученные результаты свидетельствуют о предпочтительном использовании титана при электрохимической очистке чернового индия.

Ключевые слова: индий, электроосаждение, массоперенос, перенос заряда, коэффициент диффузии, квазиобратимость, константа скорости.