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Д.В. Сокольский атындағы
«Жанармай, катализ және электрохимия институты» АҚ

Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Химия және технология сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество в глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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DISSOLUTION OF STAINLESS STEEL IN SODIUM CHLORIDE SOLUTION AT POLARIZATION BY NON-STATIONARY CURRENT

Abstract. Stainless steel is in great demand due to its mechanical strength, heat resistance, and resistance to corrosive environments. This article presents the result of a study of the electrochemical dissolution behavior of a stainless steel electrode (12X18H10T) at polarization by 50 Hz alternating current in a neutral medium (NaCl). Preliminary experiments have shown that the main processes do not take place when two stainless steel electrodes are polarized with an alternating current. It was observed that by the polarization of the “stainless steel – titanium” pair electrodes with alternating current, the alloy is intensively dissolved with the formation of iron (II) and chromium (III) ions. This is due to the “valve” properties of the oxide layer formed on the surface of the titanium electrode. A change in the value of the current density of the titanium and steel electrodes significantly affects the process of electrochemical dissolution of the alloy. At high current densities, the dissolution rate of the alloy decreases due to the deterioration of the current correcting properties of titanium. With an increase in the electrolyte concentration, the current efficiency is reduced as salt passivation occurs. A maximum value of the current efficiency of dissolution of stainless steel was observed at a current frequency of 50 Hz. High frequencies of the alternating current do not provide an adjustable duration of the anodic half-cycle for the oxidation reaction due to the frequent change of half-cycles of the alternating current. It was observed that increasing the temperature of the electrolyte reduces the current efficiency of the electrochemical dissolution of stainless steel electrodes. The effects of main electrochemical parameters on the electrolysis process have been investigated, and the optimal conditions of the alloy dissolution were established ($iT_i = 60 \text{ kA/m}^2$, $iSS = 800 \text{ A/m}^2$, $[\text{NaCl}] = 2.0 \text{ M}$, $t = 30$, $\nu = 50 \text{ Hz}$).

Key words: stainless steel, titanium electrode, alternating current, electrochemical dissolution, chromium (III) ions, iron (II) ions, current density, and current efficiency.

Introduction.

The rapid development of innovative technologies has led to the emergence of new metal alloys with special properties. 12X18H10T stainless steel is highly resistant to the harshest conditions due to the presence of various alloying elements. Stainless steels consist mainly from chromium and nickel. The corrosion resistance of austenitic stainless steels depends on high chromium chemical content. When immersed in a corrosive medium, it forms a protective oxide layer, which reduces the rate of dissolution [1]. Due to its strength, impact resistance, and corrosion resistance, it is widely used in the production of chemical, nuclear, aerospace, and heavy aircraft components [2].

The corrosive properties of stainless steel type

12X18H10T have been studied in several works [3-5]. A.V. Jarochuk and et al considered the effect of annealing and neutron irradiation parameters on the microstructure and corrosion properties of 12X18H10T stainless steel. The voltage dependence of the anode current density has been determined. Research results revealed that the corrosion resistance of steel in the chloride-containing medium depends on the intensity of redox processes on the surface of the metal, on the state of the structure at micro- and nano-levels [3]. The corrosion resistance of 12X18H10T stainless steel and new high-strength 05X22AG15H8M2F austenitic steel has been studied comparatively. Laboratory and field tests have shown that steel 05X22AG15H8M2F has higher corrosion resistance than steel 12Cr18Ni10Ti [4]. Corrosion

of 12Cr18Ni10Ti chromium-nickel steel in 2.0 M hydrochloric acid medium was studied using weight, potentiometric and voltammetric techniques. It was found that corrosion of steel in background acid solutions, as well as in the presence of inhibitors occurs in the zone of active dissolution of the metal [5].

In the next study, the corrosive properties of Cr-Mn austenitic stainless steel were studied in a sulfuric acid medium containing thiocyanate ions. In the presence of 0.01 M, NH_4SCN ammonium thiocyanate showed a thinning of the passive film, resulting in an increased corrosion rate [6].

The effect of 3.5 wt% addition of Si on the corrosion resistance of 18Cr-15N stainless steel in a nitric acid was studied via special electrochemical measurements. As a result the X1CrNiSi18-15-4 stainless steel corrodes slower than X1CrNiSi18-15-10 steel. The presence of Si in the oxide film slightly deteriorates the passivity of stainless steel and restrains the reduction process [7].

The electrochemical behavior of austenitic stainless steel (type 304) was studied by potentiodynamic polarization and weighting method in 3M sulfuric acid with 3.5% recrystallized sodium chloride. Research results displayed the absence of corrosion products and topographic changes in the steel surface layers [8].

The electrochemical dissolution of 2205 duplex stainless steel in a chloride-thiosulfate medium was studied by potentiodynamic polarization, electrochemical impedance spectroscopy, and Mott-Schottky measurements. The Cl^- and $\text{S}_2\text{O}_3^{2-}$ ions at high anodic potentials worsened the passivity of the passive film, while $\text{S}_2\text{O}_3^{2-}$ facilitated the oxidation of Cr (III) to Cr (VI) [9].

The effect of extreme stray current on the anodic dissolution and passivation of X80 steel in NaHCO_3 solution were studied by drawing polarization curves and using EIS, AFM, and SEM methods. During anodic current polarization ($i=0\sim 200\text{A/m}^2$) it was determined that the components of X80 stainless steel corrosion products were FeOOH in outer and Fe_3O_4 in inner [10].

The mechanism of growth and electrochemical properties of the oxide film on AISI 304 stainless steel were studied in fluoride media at different pH values. Fluoride concentration has a significant effect on dissolution rate and oxide film resistance relative to pH [11].

Low concentrations of thiocyanate significantly increased the active dissolution rate of austenitic and ferritic stainless steels. The effect of thiocyanate on the anodic dissolution of pure iron, pure chromium, and 430 stainless steel in 1M Na_2SO_4 solution acidified with H_2SO_4 was studied by potentiostatic and impedance measurement. Thiocyanate oxidized with sulfuric acid had little effect on the anodic dissolution of pure iron, but increased the dissolution rate of chromium to one order of magnitude, and had

a stronger effect on stainless steel type 430 [12].

The electrochemical dissolution behavior of pure iron and chromium metals were studied in various aqueous media [13-15]. The anodic dissolution of iron in H_2SO_4 and HNO_3 solutions was studied with a Pt electrode modified with polyaniline by using SECM. It is shown that $\text{Fe}(\text{OH})_2$ and (Fe_3O_4) are present on the surface of the iron electrode during the oscillating process. The results showed that the pH of the solution played a key role, and anions played an additional role in the oscillating current [13].

The effect of chloride (Cl^-) and nitrate (NO_3^-) ions during anodic dissolution in the Fe/ H_2SO_4 system was studied. At the initial stage of the passive zone, it was found that NO_3^- ions contribute to the anodic dissolution of iron by creating more active sites on the surface of the electrode at $\text{CNO}_3/\text{CCl}=1:1$. However they inhibit anodic dissolution when $\text{CNO}_3/\text{CCl}=10:1$ [14].

The dissolution mechanism of pure chromium at high current densities characteristic for electrochemical processing is studied. The mechanism of chromium dissolution was explained in detail by combining electrochemical, microscopic, and spectroscopic methods. The polishing quality has increased due to the increment in nitrate concentration and current density. 45% of the anode current used to release oxygen. With the increase of the applied potential, the valence of chromium dissolution decreased to $z = 3.5$ [15].

The results of the literature review showed that the corrosive properties of stainless steel have been studied in detail, but its electrochemical properties have not been fully explored. In our previous research, the electrochemical dissolution behavior of stainless steel was studied in the aqueous solution of sulfuric, hydrochloric, and nitric acids [16, 17]. The research results revealed that in acidic media a higher dissolution rate of stainless steel can be reached out by using alternating current electrolysis than direct current electrolysis. It was observed that under alternating current optimal conditions the current efficiency (CE) of stainless steel dissolution in sulfuric, hydrochloric, and nitric acid aqueous solution decreased accordingly: $\text{H}_2\text{SO}_4 > \text{HCl} > \text{HNO}_3$.

Methods and materials (Practical part)

Alternating current (AC) electrolysis was used to study the electrochemical dissolution behavior of a stainless steel electrode in an aqueous solution of sodium chloride. All the experiments were performed in an electrolyzer without separation of electrode space. The stainless steel (12X18H10T) plate was used as the working electrode and titanium wire was used as an additional electrode. Experiment procedure, and techniques, also elemental composition of stainless steel electrode are described in our previous work [16]. The amount of Fe^{2+} and Cr^{3+} ions formed as a result of electrolysis was determined by the photocolometric method [18-19].

The effect of current density, electrolyte concentration, electrolysis duration and AC frequency on titanium, and stainless steel electrodes on the electrochemical dissolution of stainless steel was studied. The current efficiency (CE) of the electrochemical dissolution of a stainless steel electrode was calculated for the anode half-cycle of alternating current.

Preliminary experiments have shown that when two stainless steel electrodes are polarized with an alternating current of 50 Hz, the main processes do not take place. When the second stainless steel was replaced by a titanium electrode, the current efficiency was significantly higher. This is due to the formation of an oxide layer (Ti_xO_y) with valve (semiconductor) properties at the additional titanium electrode. The titanium electrode forms an oxide layer of different compositions at different current densities and simultaneously serves as an auxiliary electrode and AC rectifier. As a result, the anodic dissolution process of the alloy is intensified [16-17, 20].

Results and discussion.

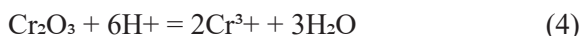
Under polarization of stainless steel electrode by AC with a frequency of 50 Hz, the following reactions can occur during the half-cycle of the anode:



The chromium component of the alloy determines the passive nature of the stainless steel. In our study, a dark green layer was formed on the surface of the stainless steel electrode. This reveals that a layer of chromium oxide is formed on the electrode surface. The formation of a passive layer of Cr_2O_3 prevents further diffusion of oxygen to the surface. The authors of the work [21] revealed that during the oxidation of pure chromium electrode to Cr^{3+} ion in NaCl solution, a thin dense oxide layer is formed at the metal-electrolyte boundary:



It was noted that the formation and growth of film occurs as a result of the migration of chromium ions towards oxygen ions. The thickness of the oxide layer generally remains approximately constant during electrolysis, since its outer side is exposed to the dissolving effect of the electrolyte by the reaction:



The electrochemical dissolution of stainless steel was studied by pairing it with titanium under alternating current polarization. When the current density at the titanium electrode increased from 20 kA/m^2 to 120 kA/m^2 , the electrochemical dissolution of stainless steel and the electrochemical formation of Fe (II) and Cr (III) ions passed through the maximum

(Figure 1). The growth in the value of CE with increasing current density can be explained by the increment in the valve properties of the oxide layers formed at the surface of titanium electrode. The total dissolution output of stainless steel at 80 kA/m^2 current density of at the titanium electrode accounted for 43.4%, for Fe (II) ions - more than 37%, and Cr (III) ions – just fewer than 7%. Further increase in the current density at the electrode reduced the CE of stainless steel dissolution. This is due to the increase in the rate of additional reactions with increasing current density.

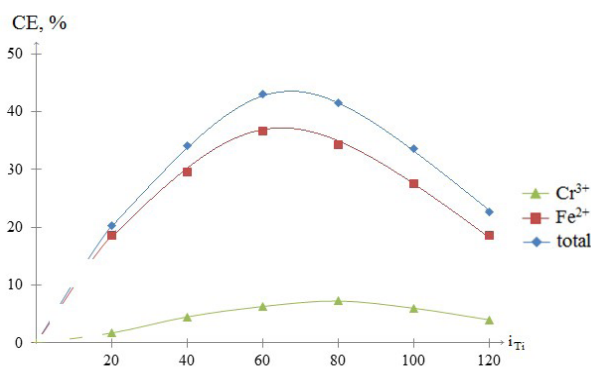


Figure 1. The influence of current density on titanium electrode on current efficiency of stainless steel electrode polarized by alternating current ($i_{SS} = 800 \text{ A/m}^2$, $[NaCl] = 1,0 \text{ M}$, $t = 0,5 \text{ h}$).

The effect of variation of the stainless steel electrode current density on the dissolution of Fe-Cr-based alloys was studied (Figure 2). At the same time, the current density in titanium was kept constant at 60 kA/m^2 . When increasing the current density at the steel electrode from 200 A/m^2 to 1200 A/m^2 , the maximum current output rate of the alloy was observed at 800 A/m^2 . The experimental data showed that the current efficiency of steel dissolution with forming iron (II), chromium (III) ions increased from 20% to 43%, and the value of CE decreased with further increase in current density. At higher current densities, due to the increase in the rate of additional reactions, the values of the dissolution rate of the alloy decreased.

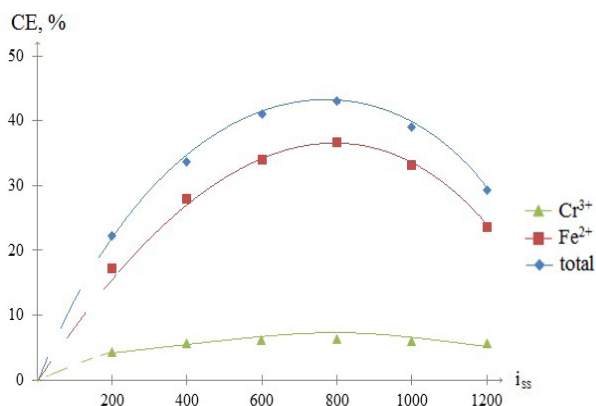


Figure 2. The influence of current density on

stainless steel electrode on current efficiency of alternating current polarized stainless steel electrode ($i_{Ti} = 60 \text{ kA/m}^2$, $[\text{NaCl}] = 1,0 \text{ M}$, $t = 0,5 \text{ h}$).

The effect of changes in the concentration of sodium chloride was studied in the range of 0.5-3.0 M at a current density of stainless steel electrode - 800 A/m^2 , and titanium electrode - 60 kA/m^2 (Figure 3). With an increase in the concentration of sodium chloride solution to 2.0 M, an intensive dissolution of the stainless steel electrode was observed. The maximum current efficiency of the stainless steel electrode dissolution reached - 57.28%, and accounted for Fe (II) ions - 49.5%, Cr (III) ions - 7.78% respectively. With further increase in the electrolyte concentration, the current efficiency is reduced as salt passivation occurs

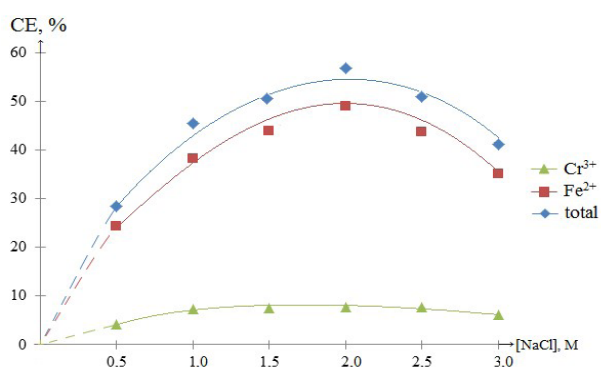


Figure 3. Influence of solution concentration on current efficiency polarized by alternating current stainless steel electrode dissolution ($i_{SS} = 800 \text{ A/m}^2$, $i_{Ti} = 60 \text{ kA/m}^2$, $t = 0,5 \text{ h}$).

The electrochemical dissolution properties of an AC polarized stainless steel electrode were studied at the electrolyte temperature of 20-60°C (Figure 4). As the temperature of the solution increases, the current output of Fe (II) and Cr (III) ions initially increases and then gradually decreases. During the electrolysis, it was shown that the current efficiency of the stainless steel electrode at 30°C electrolyte temperature exceeds 49%.

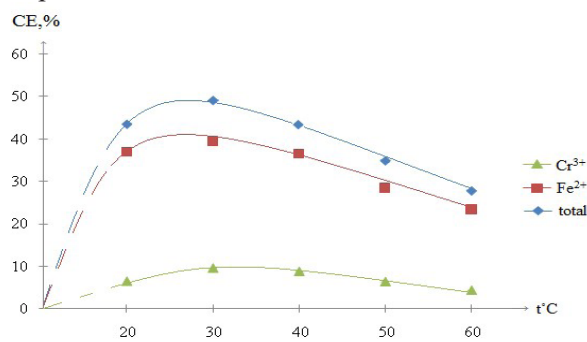


Figure 4. Influence of solution temperature on current efficiency on stainless steel electrode dissolution polarized by alternating current ($i_{SS} = 800 \text{ A/m}^2$, $i_{Ti} = 60 \text{ kA/m}^2$, $[\text{NaCl}] = 1,0 \text{ M}$, $t = 0,5 \text{ h}$).

The influence of the alternating current frequency on the electrochemical dissolution behavior of stainless steel electrodes in the range of 50–250 Hz was studied (Figure 5). With an increase in the current frequency, the dissolution current efficiency of the stainless steel electrode decreases, and the dissolution rate slows down at a frequency of 250 Hz. The maximum current efficiency of stainless steel was observed at 50 Hz AC. As the current frequency increased, the current output value decreased up to 5.6%. High frequencies of the alternating current do not provide an adjustable duration of the anodic half-cycle for the oxidation reaction of iron and chromium ions due to the frequent change of half-cycles of the alternating current.

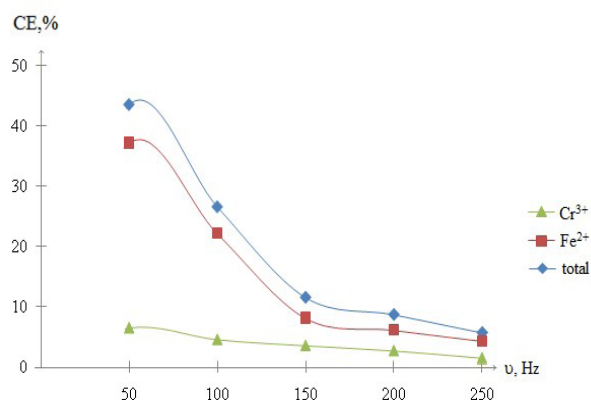


Figure 5. The effect of alternating current frequency on the current efficiency on of stainless steel electrode dissolution by polarized alternating current ($i_{SS} = 800 \text{ A/m}^2$, $i_{Ti} = 60 \text{ kA/m}^2$, $[\text{NaCl}] = 1,0 \text{ M}$, $t = 0,5 \text{ h}$).

Conclusion.

The electrochemical dissolution behavior of stainless steel electrode polarized with 50 Hz alternating current in sodium chloride solution of was systematically studied. Research results revealed that under the polarization of a pair of electrodes “stainless steel – titanium” with alternating current, the stainless steel is intensively dissolved with the formation of iron (II) and chromium (III) ions. The dependence of the electrolysis process on the change in the current density on the stainless steel and titanium electrodes, the concentration of sodium chloride, the temperature of the electrolyte, and the frequency of the alternating current have been established. It was found that a change in the value of the main electrochemical parameters significantly affects the process of electrochemical dissolution of the alloy. As a result of study the optimal conditions of the alloy dissolution were established ($i_{Ti} = 60 \text{ kA/m}^2$, $i_S = 800 \text{ A/m}^2$, $[\text{NaCl}] = 2.0 \text{ M}$, $t = 30$, $\nu = 50 \text{ Hz}$). In the effective conditions of AC electrolysis the current efficiency of SS dissolution constituted 43.5%, and for iron (II) and chromium (III) accounted for 37% and 6.5% respectively.

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ТОТ БАСПАЙТЫН БОЛАТТЫҢ НАТРИЙ ХЛОРИДІ ЕРІТІНДІСІНДЕ СТАЦИОНАРЛЫ ЕМЕС ТОҚТАРМЕН ПОЛЯРИЗАЦИЯЛАУ КЕЗІНДЕ ЕРУІ

Аннотация. Тот баспайтын болат механикалық беріктігі, ыстыққа төзімділігі, коррозиялық орталарға тұрақтылығына байланысты үлкен сұранысқа ие. Ұсынылған мақалада жиілігі 50 Гц айнымалы токпен поляризацияланған тот баспайтын болат (12Х18Н10Т) электродының бейтарап ортадағы (NaCl) электрохимиялық еру ерекшелігі зерттелген. Алдын ала жүргізген тәжірибе нәтижесі тот баспайтын болат электродының екеуін жиілігі 50 Гц айнымалы токпен поляризациялағанда негізгі үдерістердің жүрмейтіндігін көрсетті. «Тот баспайтын болат – титан» электродтар жұбын айнымалы токпен поляризациялау барысында құйманың темір (II) және хром (III) иондарын түзе қарқынды еритіндігі анықталды. Бұл титан электродының бетінде түзілген әртүрлі құрамдағы оксидті қабаттың «вентильдік» қасиетіне байланысты. Титан және болат электродтарындағы ток тығыздықтарының мәнін өзгерту құйманың электрохимиялық еру процесіне елеулі әсер етті. Жоғары ток тығыздықтарында титанның ток түзеткіштік қасиетінің нашарлауына байланысты құйманың еру жылдамдығы төмендейді. Электролит концентрациясын жоғарылатқанда тұзды пассивация орын алуына орай ток бойынша шығымы төмендеді. Ток жиілігінің 50 Гц мәнінде тот баспайтын болат еруінің ток бойынша максималды шығымы байқалды. Айнымалы ток жиілігінің жоғары шамасында периодтар жиі өзгертіндіктен, анодтық жартылай периодта құйма құрамындағы темір және хром иондарының тотығу үдерісіне қажет анодтық жартылай период ұзақтығы қамтамасыз етілмейді. Электролит температурасын арттырғанда тот баспайтын болат электродының электрохимиялық еруінің ток бойынша шығымы төмендейтіндігі байқалды. Айнымалы токпен поляризацияланған тот баспайтын болат электродының бейтарап ортадағы электрохимиялық еру үрдісіне негізгі электрохимиялық параметрлердің әсерлері зерттеліп, құйма еруінің тиімді жағдайлары қалыптастырылды ($i_{Ti} = 60 \text{ кА/м}^2$, $i_B = 800 \text{ А/м}^2$, $[NaCl] = 2.0 \text{ М}$, $t = 30$, $v = 50 \text{ Гц}$).

Түйін сөздер: тот баспайтын болат, титан электроды, айнымалы ток, электрохимиялық еру, хром (III) иондары, темір (II) иондары, ток тығыздығы, ток бойынша шығым.

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РАСТВОРЕНИЕ НЕРЖАВЕЮЩЕЙ СТАЛИ В РАСТВОРЕ ХЛОРИДА НАТРИЯ ПРИ ПОЛЯРИЗАЦИИ НЕСТАЦИОНАРНЫМИ ТОКАМИ

Аннотация. Нержавеющая сталь пользуется большим спросом благодаря своей механической прочности, жаростойкости, устойчивости к коррозионным средам. В данной статье приведены результаты исследования особенности электрохимического растворения электрода из нержавеющей стали (12Х18Н10Т) при поляризации переменным током частотой 50 Гц в нейтральной среде (NaCl). Предварительные эксперименты показали, что при поляризации двух электродов из нержавеющей стали переменным током с частотой 50 Гц основные процессы не протекают. При поляризации пары электродов «нержавеющая сталь-титан» переменным током установлено, что сплав интенсивно растворяется с образованием ионов железа (II) и хрома (III). Это связано с «вентильными» свойствами оксидного слоя разного состава, образующегося на поверхности титанового электрода. Установлено, что изменение значения плотности тока титановом и стальном электроде существенно влияет на процесс электрохимического растворения сплава. При высоких плотностях тока скорость растворения сплава снижается из-за ухудшения тококорректирующих свойств титана. При увеличении концентрации электролита выход по току снижался по мере возникновения солевой пассивации. Высокое значение выхода по току растворения нержавеющей стали наблюдалось при частоте тока 50 Гц. Высокие частоты переменного тока не обеспечивают необходимую продолжительность анодного полупериода для протекания реакции окисления ионов железа и хрома из-за частой смены полупериодов переменного тока. Было обнаружено, что выход по току электрохимического растворения электрода из нержавеющей стали уменьшается с увеличением температуры электролита. Исследовано влияние

основных электрохимических параметров на процесс электрохимического растворения электрода из нержавеющей стали при поляризации переменным током в нейтральной среде, и установлены оптимальные условия растворения сплава ($i_{Ti} = 60 \text{ кА/м}^2$, $i_C = 800 \text{ А/м}^2$, $[\text{NaCl}] = 2.0 \text{ М}$, $t = 30$, $v = 50 \text{ Гц}$).

Ключевые слова: нержавеющая сталь, титановый электрод, переменный ток, электрохимическое растворение, ионы хрома (III), ионы железа (II), плотность тока, выход по току.

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CHEMICAL ACTIVATION OF THE SURFACE OF THE BURNT ROCK BY VARIOUS MODIFIERS

Abstract. This article presents the results of experimental studies of the process of grafting humic acids and their functional derivatives on modified and unmodified surfaces of burnt rock. It is shown that the inoculation of humic acids on inorganic carriers is more successful with the preliminary activation of the surface of burnt rocks.

The acidic and sorption properties of grafted surfaces of humic sorbents with respect to heavy metal ions have been studied. It is shown that many of the modifying additives on the surface of the inorganic carrier can increase these properties by 1.2-2.0 times in comparison with the original rock.

Surface modification was carried out by means of impregnation with water solutions of the modifier and subsequent heat treatment; heating at boiling point in water solutions with subsequent heat treatment of precipitation.

The interaction of modifiers with the surface occurs by the type of ion exchange for silanol or aluminol groups with weak acidity. It is also possible to connect via hydrogen bonds or dipole-dipole and induction interactions. It was found that the degree of grafting of humic compounds is significantly higher on the activated surfaces of the burnt rock.

The content of the modifier on the surface of the carrier is 10-40%, depending on the experimental conditions and the nature of the modifier. The most active modifiers are sodium phosphate and potassium carbonate.

The kinetics of the interaction of the surface of the burnt rock with potassium carbonate of different

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