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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
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АО «Институт топлива, катализа и
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STUDY OF ON THE ELECTROCHEMICAL BEHAVIOR OF TITANIUM IN ACIDIC BROMIDE SOLUTION BY RECORDING THE POTENTIODYNAMIC POLARIZATION CURVES

Abstract. The electrochemical dissolution of titanium in a potassium bromide solution acidified with a sulfuric acid solution by recording cyclic and anodic potentiodynamic polarization curves was studied for the first time. The influence of the sulfuric acid and potassium bromide solutions concentration on the titanium electrode oxidation was considered.

A polarization curve was performed in 1.0 M potassium bromide solution in the presence of 0.5 M sulfuric acid and the titanium oxidation anodic maximum was recorded. To explain the titanium dissolution characteristics during the polarization by industrial alternating current, cyclic anodic-cathodic (CAC) and cyclic cathode-anodic (CCA) polarization curves of the titanium electrode in potassium bromide sulfate solution were recorded. At the CAC voltammogram, when the titanium potential is shifted to the anode direction, titanium oxidation current is recorded at potentials from “plus” 1.3 V to “plus” 2.3 V. In the cathode direction of the CCP voltammogram curve between potentials “minus” 0.7–1.0 V, a hydrogen evolution current is observed. The dependence of anodic potentiodynamic polarization curves on the concentration of sulfuric acid and potassium bromide solutions was studied, in both cases an increase in the titanium oxidation maximum current was observed, a mechanism was also established and the reactions sequence proceeding on the electrode was calculated.

Studies have shown that electrode reactions occur by a complex mechanism.

Keywords: titanium, potassium bromide, sulfuric acid, electrode, potentiodynamic polarization curves, oxidation current.

Titanium can be called modern techniques and industry metal since most of its part is used for space, aero- and hydro technologies needs. In its turn, titanium (IV) oxide is the main titanium industry product. The classic methods of its production are a complex process that involves the production of titanium sulfate, tetrachloride and bromide solutions for obtaining titanium minerals called ilmenite, leucoxene and rutile. It is very difficult to extract these salts directly from titanium residues (scrap) since titanium is a highly corrosion-resistant metal with high-melting temperature in many aqua environments [1]. It is important to determine the electrochemical dissolution laws, mechanisms and kinetics of corrosion-resistant metals in various aqueous solutions.

M.S. Amrutha used a four-stage mechanism to determine chemical constants by investigating of Ti in varying concentrations of hydrofluoric acid (0.01 M÷1.0 M) through applying potentiodynamic polarization and electrochemical impedance spectroscopy methods [2]. F. Fasmin presented a kinetic model of titanium electrode anode dissolution in 0.1 M hydrofluoric acid in active and passive regions [3]. A.B. Bayeshov et al. have considered electrochemical dissolution of titanium in the sulfur, hydrochloric acid and phosphoric acid medium in the solution with fluoride ions in the presence of alternating current [4-9]. The study result shows that titanium electrode dissolves with high current efficiency in acidic solutions containing fluoride ions.

U. Tetsuya studied the electrochemical dissolution of titanium electrode in the TMHA-Tf_2N ionic liquid. It was determined that no dissolution of titanium was observed during the anodic potential sweep up to +6.0 V due its oxide film formation. Prior to electrochemical experiments, more mechanical smoothing of titanium surface led to the shift of the titanium potential immersed in the electrolyte to a negative value. In this case, the anode titanium dissolution in the zone with the potential value "minus" 0.95 ÷ "plus" 1.6 V was determined [10].

D. Baehre considered the electrochemical dissolution behavior of pure titanium electrode, TiMaAl6V4 and intermetallic Ti60Al40 (γ-TiAl) in potassium bromide-, sodium chloride-, sodium nitrate aqueous solutions at different pH-values. During the study, the methods of cyclic voltammetry, linear sweep voltammetry polarization and chronoamperometry were performed. As a result, a positive effect of halide compounds on the titanium electrode dissolution and its alloys was determined [11].

Literature data analysis shows that electrochemical properties of titanium in bromide acidic environment have not been studied yet. Having investigated titanium (III) ions oxidation mechanism, A.B. Bayeshov and G.M. Iztileuov show that titanium (III) ions oxidation occurs as a result of the hydrolysis process at high temperatures and can be met either in $[Ti(H_2O)_6]Cl_3$ or $[Ti(H_2O)_6]SO_4$ form in the acidic medium. As a result of the study, at 333 K temperature, an increase of titanium (III) ion oxidation wave in a sulfuric acid concentration of 300 g/l was observed, and the most maximum current negative value was "minus" 0.8 V in the graphite electrode and "minus" 0.02 V in lead electrode [12].

Y. Jiang and Y. Wu investigated the electrochemical reaction mechanism of titanium alloy in neutral (NaCl) aqua environment performed by polarization curves. It was defined the formation of titanium intermediate oxide shell through oxidizing during its anode dissolution in the sodium chloride solution [13].

The advantage of the titanium anode oxidation process as a coating in materials surface covering in the biomaterials production has been shown in a number of studies [14, 15]. E.A. Kornyushova et al. studied electrochemical properties of titanium and platinum electrodes in dicarboxylic amino acids. Due to the implementation of the surface reactions in the titanium electrode, the oxide shell has been grown; thereby the existence of its adsorbing properties has been determined [16]. Young-Taeg Sul et al. discovered the electrochemical properties of titanium biomaterials in acetic acid, phosphoric acid, calcium hydroxide and sodium hydroxide, and showed that the anode voltage stress (dV/dt) in acidic electrolyte was higher than in alkaline electrolyte [17].

In the given work, electrochemical properties of titanium were studied by recording potentiodiodynamic polarization curves in potassium bromide aqueous solution acidified by sulfuric acid solution.

The polarization measurement was performed using potentiostat "Autolab". Voltampereometric studies were conducted in three-electrode thermostated electrolysis cells. A silver-chloride electrode was used as a reference electrode, and platinum cord as an auxiliary electrode. The outer surface of titanium covered by teflon with a diameter of 2 mm and used as a working electrode.

Before each experiment, the titanium electrode was rinsed with distilled water and smoothed in 500 μm and 200 μm sandpaper material, defatted and washed thoroughly with filter paper. Different concentrations of KBr and H_2SO_4 aqueous solutions were used as electrolytes.

When the titanium electrode is polarized by direct current in the Galvanometric case, its surface is passivated by oxidized layers and electrode dissolution by forming titanium ions is accompanied by very low current efficiency. Results of our previous research show, titanium electrode intensively dissolved in the sulfuric acid bromide solution polarized by the industrial alternating current has been observed [18, 19].

The titanium electrode properties in potassium bromide solution were investigated first in potassium bromide and sulfuric acid solution, subsequently by recording anodic and cyclic potentiodynamic polarization curves in mixed solutions of potassium bromide and sulfuric acid.

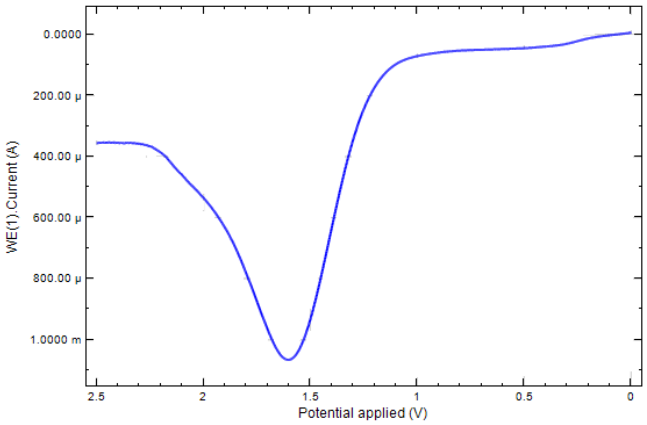
Anodic polarization curves were taken to determine the titanium electrode specificity to dissolve in potassium bromide and sulfuric acid.

In the potentiodynamic polarogram taken in the anode direction the titanium oxidation wave in potassium bromide or sulfuric acid solutions was not recorded.

In subsequent studies, 1.0 M potassium bromide solution was dissolved in 0,5 M sulfuric acid solution and polarization curve "potential-current density" was recorded. In this solution, it is possible to notice that the anodic titanium oxidation current was recorded in the anode potentiodynamic curve.

In the cyclic mode, potassium bromide and sulfuric acid solutions were recorded in cycles between "plus" 2.5 V and "minus" 1.0 V in order to detect the specificity of processes occurring in the titanium electrode polarized by industrial alternating current with the frequency of 50 Hz.

When the CAC potential shifts in the anode direction, the maximum oxidation current between "plus" 1.3 V ÷ "plus" 2.3 V potential is recorded on the polarogram (Figure 1). In the polarogram of cathodic direction there were no significant oxidation waves, the hydrogen evolution was detected at "minus" 0.7 V and at negative potentials.



[KBr]=1M; [H_2SO_4]=0.5M; ν=50мВ/с

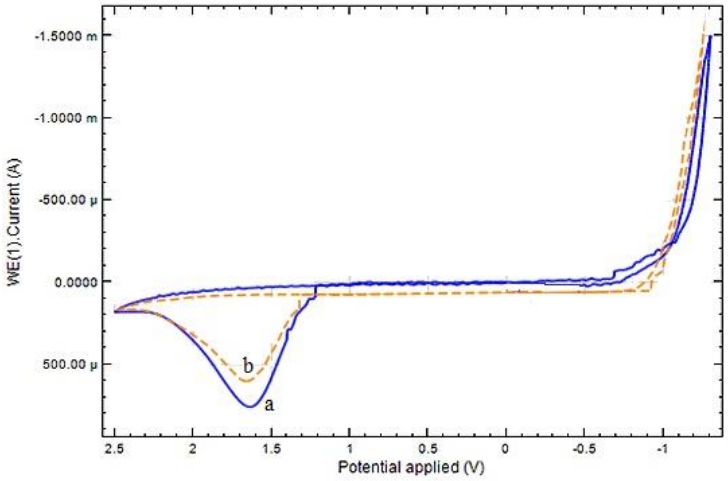
Figure 1 - Anodic potentiodiodynamic polarization curve of titanium electrode in acidic bromide solution

In the acidified potassium bromide solution, titanium oxidation in the anode direction can be described by the reaction. In the cathodic direction, mainly hydrogen ion discharge occurs (reaction 2).

$Ti - 3e^- \rightarrow Ti^{3+}$ $E^0 = -1{,}21V$ (1)

$2H^+ + 2e^- = H_{2\uparrow}$ (2)

In the polarization curve taken in the CCA direction, the main processes occurring in the electrode are repeated (Figure 2). The CCA polarogram cathode direction shows that beginning from "minus" 0.7-1.0 V potential hydrogen gas division is observed and only the titanium oxidation maximum value decreases (Fig. 1, b-curve).

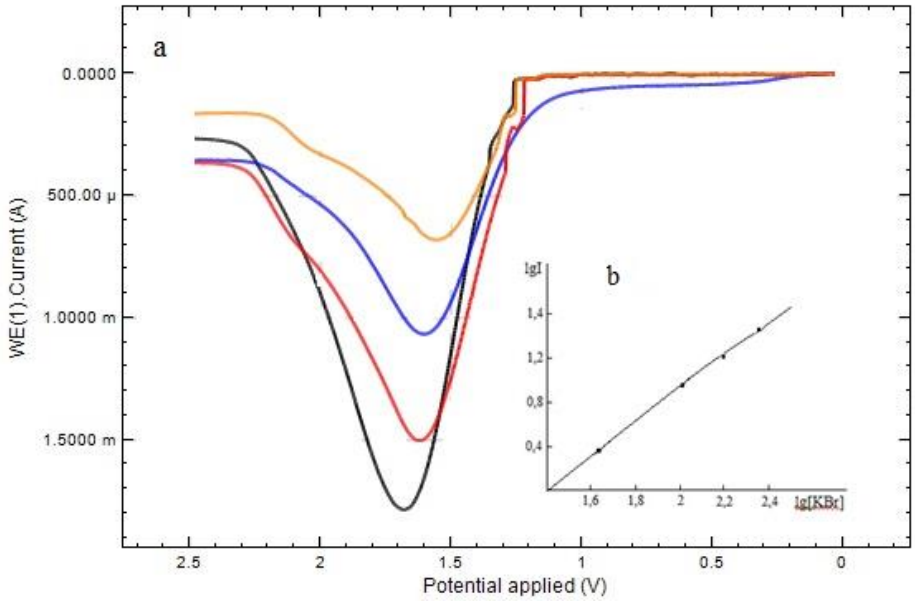


[KBr]=1.0 M; [H_2SO_4]=0.5 M; ν=50мВ/с

Figure 2 - Cathodic-anodic (a) and anodic-cathodic (b) cyclic voltammogram of titanium electrode in the acidic bromide solution

The potassium bromide concentration effect on titanium electrode anodic potentiodiodynamic polarization curves is shown in Figure 3 (a). Increasing the potassium bromide solution concentration can significantly increase the oxidation current height. According to the experiment results, dependence taken on lg[KBr] – lgi coordinate is straight linear (b). Table 1 was completed based on this dependence result and the reaction rate for bromide ions was 0.2.

Logarithmic dependence should be straight linear to determine the reaction sequence. The reaction sequence was mathematically determined [20].



[KBr]=0.5-2.0 M; [H_2SO_4]=0.5 M; ν=50мВ/с

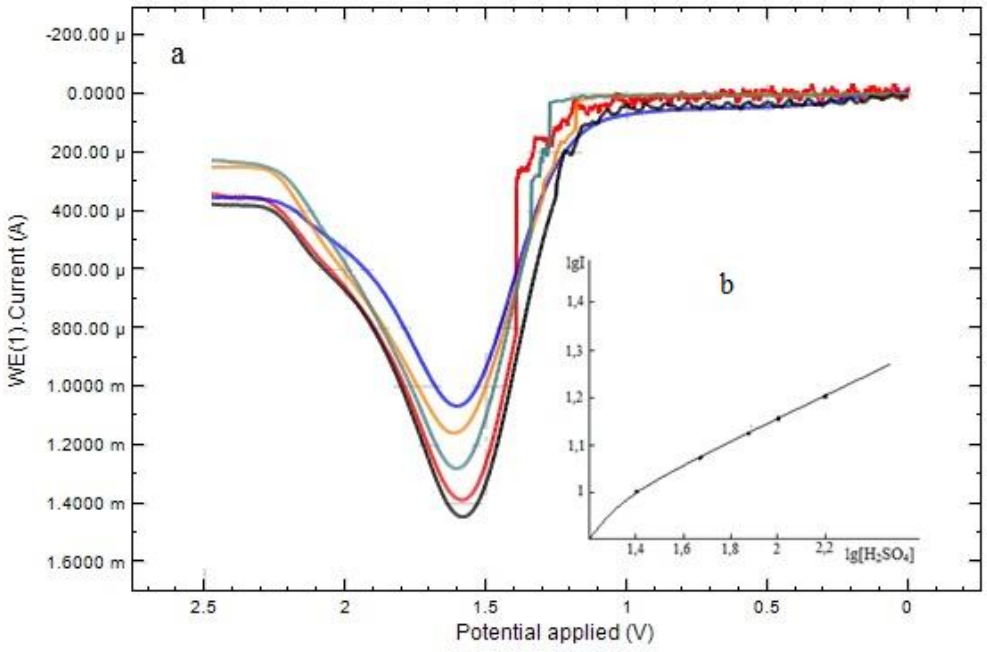
Figure 3 - Dependence of titanium electrode anodic potentiodynamic polarization curves on the anode maximum of potassium bromide concentration (a) and the lg*i*-lgC_{KBr} dependence on potassium bromide concentration (b)

Table 1 - The electrode reaction order of bromide ions occurring during electrolysis

№	C_{el}	lgC=x	i	y=lgi	x·y	x^2
1	0.5	-0.301	65	1.8	-0.5418	0.29
2	1.0	0	105	2.0	0	0
3	1.5	1.76	150	2.2	0.3872	0.1499
4	2.0	0.301	180	2.25	0.6772	0.4586
Σ	-	0.176	-	8.25	0.5226	0.8985

$$b = \frac{n\sum x \cdot y - \sum x \cdot \sum y}{n\sum x^2 - \left(\sum x\right)^2} = \frac{4 \cdot 0.5226 - (0.176 \cdot 8.25)}{4 \cdot 0.8925 - (0.176)^2} = 0.17 \approx 0.2$$

Figure 4 (a) shows the titanium electrode dependence on the anodic potentiodynamic polarization curves and on the sulfuric acid concentration. Increasing the sulfuric acid concentration can significantly increase the oxidation current height rightfully. According to the experiment results, the dependence taken on the lg[H_2SO_4] – lgi coordinate is straight linear (b). Increasing the sulfuric acid concentration increases the oxidation current height evenly. Based on this dependence result, Table 2 was completed and the fact that the reaction rate for sulfuric acid was 0.04 was detected.



[KBr]=1.0 M; [H_2SO_4]=0.25-1.50 M; ν=50мВ/с

Figure 4 - The dependence of sulfuric acid concentration on the anode maximum at titanium electrode anodic potentiodynamic polarization curves (a) and the dependence the logarithms of sulfuric acid concentration and the maximum current height (b)

Table 2 - The sequence of electrode reactions on sulfuric acid during electrolysis

№	C_{el}	lgC=x	I	y=lgi	x·y	x^2
1	0.25	-0.602	105	2.02	-1.216	1.4800
2	0.50	-0.302	115	2.06	-0.620	0.3800
3	0.75	-0.120	130	2.11	-0.250	0.0625
4	1.00	0	140	2.15	0	0
5	1.50	0.176	145	2.16	0.380	0.1400
Σ	-	-0.847	-	10.5	-1.706	2.0600

$$b = \frac{n\sum x\cdot y - \sum x\cdot \sum y}{n\sum x^2 - \left(\sum x\right)^2} = \frac{5\cdot(-1.706) - (-0.847\cdot 10.5)}{5\cdot 2.06 - (-0.847)^2} = 0.04$$

Summing up, the electrochemical properties of titanium in acid bromide solution were first studied by recording potentiodynamic polarization curves. No dissolution of titanium electrode was observed during the anodic polarization in 1.0 M potassium bromide and 0.5 M sulfuric acid solutions separately from each other. With a view to increasing the electrochemical dissolution process, polarization curves were taken in 1.0 M potassium bromide solution in the presence of 0.5 M sulfuric acid and the maximum oxidation rate recording was observed. In this case, the mechanisms and kinetics of electrode reactions were determined. The titanium electrode was studied by recording polarization CAC and CCA in potassium bromide and sulfuric acid solutions. The specificity of titanium electrode dissolution polarized with alternating current of 50 Hz is determined. The dependence of anodic potentiodynamic polarization curves on the sulfuric acid concentration and the potassium bromide concentration was investigated and in both cases the current maximum increase was observed and the reactions sequence was determined.

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ТИТАННЫҢ ҚЫШҚЫЛДЫ БРОМИДТІ ЕРІТІНДІСІНДЕГІ ЭЛЕКТРОХИМИЯЛЫҚ ҚАСИЕТІН ПОТЕНЦИОДИНАМИКАЛЫҚ ПОЛЯРИЗАЦИЯЛЫ ҚИСЫҚТАР ТҮСІРУ АРҚЫЛЫ ЗЕРТТЕУ

Аннотация. Күкірт қышқылымен қышқылданған калий бромиді ерітіндісінде титанның электрохимиялық еру заңдылықтары циклді және анодты потенциодинамикалық поляризациялық қисықтар түсіру арқылы алғаш рет зерттелді. Титан электродының анодтық тотығу процесіне күкірт қышқылы мен калий бромиді ерітінділерінің концентрация әсері қарастырылды.

Құрамында 1,0 М калий бромиді және 0,5 М күкірт қышқылы бар ерітіндіде поляризациялық қисықтар түсірілді және бұл кезде анодты тотығу максимумының тіркелгендігі көрсетілді.

Өндірістік жиіліктегі айнымалы токпен поляризацияланған титанның еру ерекшелігін түсіндіру үшін титан электродының калий бромиді мен күкірт қышқылы бар ерітінділердегі циклді анод-катод (ЦАК), циклді катод-анод (ЦКА) бағыттарында поляризациялық қисықтар түсірілді. ЦАК полярограммасында титан потенциалын анод бағытына қарай ығыстырғанда «плюс» 1,3 В ÷ «плюс» 2,3 В потенциалы аралығында титанның тотығу тогы тіркелді. ЦКА полярограммасының катод бағытында «минус» 0,7-1,0 В потенциалдарынан бастап, сутек газының бөліну тогы байқалды.

Анодты потенциодинамикалық поляризациялық қисықтарға әртүрлі күкірт қышқылы және калий бромиді концентрацияларының әсері зерттеліп, екі жағдайда да титанның анодты еру ток максимумдарының жоғарылайтындығы анықталды, сонымен қатар, электродта жүретін реакциялардың реті есептеліп, механизмі анықталды.

Зерттеу нәтижесінде титан электродында жүретін реакциялардың өте күрделі механизммен іске асатындығы көрсетілді.

Түйін сөздер: титан, калий бромиді, күкірт қышқылы, электрод, потенциодинамикалық поляризациялық қисықтар, тотығу тогы.

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ИССЛЕДОВАНИЕ ЭЛЕКТРОХИМИЧЕСКОГО ПОВЕДЕНИЯ ТИТАНА В КИСЛЫХ БРОМИДНЫХ РАСТВОРАХ МЕТОДОМ СНЯТИЯ ПОТЕНЦИОДИНАМИЧЕСКИХ ПОЛЯРИЗАЦИОННЫХ КРИВЫХ

Аннотация. Впервые исследовано электрохимическое растворение титана в растворе бромида калия, подкисленном раствором серной кислоты методом снятия циклических и анодных потенциодинамических поляризационных кривых. Рассмотрено влияние концентрации растворов серной кислоты и бромида калия на процесс окисления титанового электрода.

Снята поляризационная кривая в 1,0 М растворе бромида калия в присутствии 0,5 М серной кислоты и зафиксирован анодный максимум окисления титана. Для объяснения особенностей растворения титана при поляризации промышленным переменным током сняты циклические анодно-катодные (ЦАК) и циклические катодно-анодные (ЦКА) поляризационные кривые титанового электрода в сернокислом растворе бромида калия. На ЦАК полярограмме при смещении потенциала титана в анодном направлении регистрируется ток окисления титана при потенциалах от «плюс» 1,3 В до «плюс» 2,3 В. В катодном направлении полярограммы ЦКА кривой между потенциалами «минус» 0,7-1,0 В наблюдается ток выделения водорода. Исследована зависимость анодных потенциодинамических поляризационных кривых от концентрации раствора серной кислоты и бромида калия, в обоих случаях наблюдается увеличение максимума тока окисления титана, также установлен механизм и рассчитан порядок реакций, протекающих на электроде.

Исследования показали, что электродные реакции протекают по сложному механизму.

Ключевые слова: титан, бромид калия, серная кислота, электрод, потенциодинамические поляризационные кривые, ток окисления.

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