

Anodic Behavior of a Titanium–Aluminum Hybrid Electrode: Formation of Hydroxide-Oxide Compounds

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Received August 6, 2016

Abstract—The electrochemical behavior of a titanium–aluminum hybrid electrode in aqueous solutions of electrolytes containing halide ions (F^- and Cl^-) was studied. The effects of current density, solution composition, and ratio of the working surface area of titanium and aluminum on the anodic dissolution rate of a Ti–Al hybrid electrode and its electrochemical characteristics were revealed. The joint anodic dissolution of aluminum and titanium in the aqueous media under study made it possible to obtain precursors of the highly disperse oxide system Al_2O_3 – TiO_2 . Data of X-ray and electron-microscopic analysis confirmed the results obtained.

Keywords: anodic dissolution, halide ions, hybrid electrode, aluminum, titanium, oxide system

DOI: 10.1134/S2070205117060090

INTRODUCTION

Aluminum and titanium disperse oxides are used to obtain highly efficient catalysts and heat-resistant catalyst carriers, highly selective sorbents, polymeric fillers, and new functional coatings and materials [1]. Currently, ultradispersed and nanomodified titanium dioxide, which is used in solar cells [2, 3]; chemical and biological sensors [4, 5]; polymeric membrane materials [6, 7]; special ceramics, including medical biomaterials [8]; photocatalysts [9, 10]; etc., is attracting special attention.

Nanostructured TiO_2 can be obtained via oxidation of metallic titanium with various chemical reagents. TiO_2 nanorods, for example, have been obtained through oxidation of a titanium plate in H_2O_2 solution (30 wt %) at 353 K for 72 h [11]. Control of a titanium dioxide crystal structure is possible via addition of sodium halides NaX ($X = F^-$ and Cl^-). Anodic oxidation of titanium in an electrolyte containing fluoride ions makes it possible to obtain TiO_2 nanostructured nanotubes, the parameters of which can be controlled by varying the oxidation conditions [12, 13].

Currently, the synthesis of complex oxide–hydroxide highly dispersive systems, for example, aluminum–titanium oxides, which are precursors for new promising ceramic and composite materials with improved functional properties, is an important subject [14–17].

The electrochemical methods are proposed to be used to obtain such systems [18, 19]. The advantages of this approach are composition stability of the nano-

dispersed compounds with reproducible properties and the possibility of efficiently controlling the shape, size, morphology, phase, and chemical composition of the particles.

The aim of this work is to reveal the electrochemical behavior of a hybrid titanium–aluminum electrode under anodic polarization conditions in aqueous solutions containing chloride and fluoride ions, as well as to study the formation of oxide–hydroxide compounds. The data obtained will make it possible to solve the problem of obtaining precursors of the highly disperse Al_2O_3 – TiO_2 system via anodic dissolution of a hybrid titanium–aluminum electrode in aqueous halide-containing solutions.

EXPERIMENTAL

Polarization measurements were performed in a standard electrolytic cell with a platinum electrode as an auxiliary one and two saturated EVL-1 silver chloride electrodes ($E = 0.222$ V) as a reference one under standard conditions (101.3 kPa and 298 K) and natural aeration in the galvanostatic regime. The interval of the current-density change was 0.1 mA/cm² and potential delay was 30 s. A hybrid electrode consisting of rigidly electrically contacting A5 aluminum (purity 99.50%) and VT 1-0 titanium plates (98.33%) was used as a working sample (anode). The distance between these metal plates (with a total working surface of 9 cm²) was 20 mm. The ratio between the aluminum working surface and that of titanium was changed during the experiments ($S(Al) : S(Ti) = 1 : 1$,

2 : 1, and 5 : 1). The working electrode was mechanically polished with a diamond paste, degreased with ethanol, and washed with bidistilled water before the experiment. Polarization of the working electrode was provided on a Zive SP 2 electrochemical station. Simultaneous measurement of potentials for Al and Ti in the hybrid electrode was carried out on P-30IM potentiostat–galvanostats.

Electrochemical dissolution of the Al–Ti hybrid electrode was performed in a 400-mL coaxial electrochemical reactor at constant current with a MATRIX source (MPS-7101 model). A 1.0 M NaCl chemically pure aqueous solution containing 0.01, 0.025, and 0.05 mol/L of hydrofluoric acid as an additive was used as an electrolyte. The electrolysis regime's amperage was 5.5–6 A, the voltage was 10–30 V, and the electrolysis time was 45–80 min.

The central electrode (cathode) in the coaxial reactor was a wire 1.5 mm in diameter made of 12X18H10T steel. A Ti–Al hybrid electrode consisting of two titanium and two aluminum plates, which alternated and had the same pair sizes, was used as a soluble electrode (anode). The precipitate obtained during electrolysis process for crystallization was kept in the mother liquor for 24 h, filtered, and dried under 80°C.

X-ray diffraction analysis was carried out according to the standard procedure on a D2 PHASER diffractometer with monochromatized $\text{CuK}\alpha$ radiation (30 kV and 10 mA) in the step-scan mode (scanning step $2\theta = 0.05^\circ$, exposure time at the point of 1 s, and acquisition interval $2\theta = 3^\circ\text{--}130^\circ$). Samples for analysis were prepared by pressing the test material into a standard quartz glass cuvette; a sample was rotated during acquisition in its own plane at a rate of 5 rpm. Calculation of interplanar distances of the diffraction reflexes was automatically carried out with a DIFFRAC. EVA software package. Identification of crystalline phases was performed via comparison of experimental interplanar distances and relative intensities with those of the reference. The quantitative amount of precipitate phase composition was calculated through the corundum numbers method.

Microscopic studies of the anode surface after polarization were carried out on an EVEX Mini SEM SX-3000 scanning electron microscope with elemental analysis (15 kV and 10 μA).

RESULTS AND DISCUSSION

The corrosion resistance of titanium is significantly higher than that of all stainless steels and aluminum, even in seawater and hot concentrated chloride solutions, due to the surface oxide film. Titanium in halide solutions is in a stable passive state and acts as a cathode relative to other metals, in particular, to aluminum. Contact with titanium accelerates the aluminum corrosion [20].

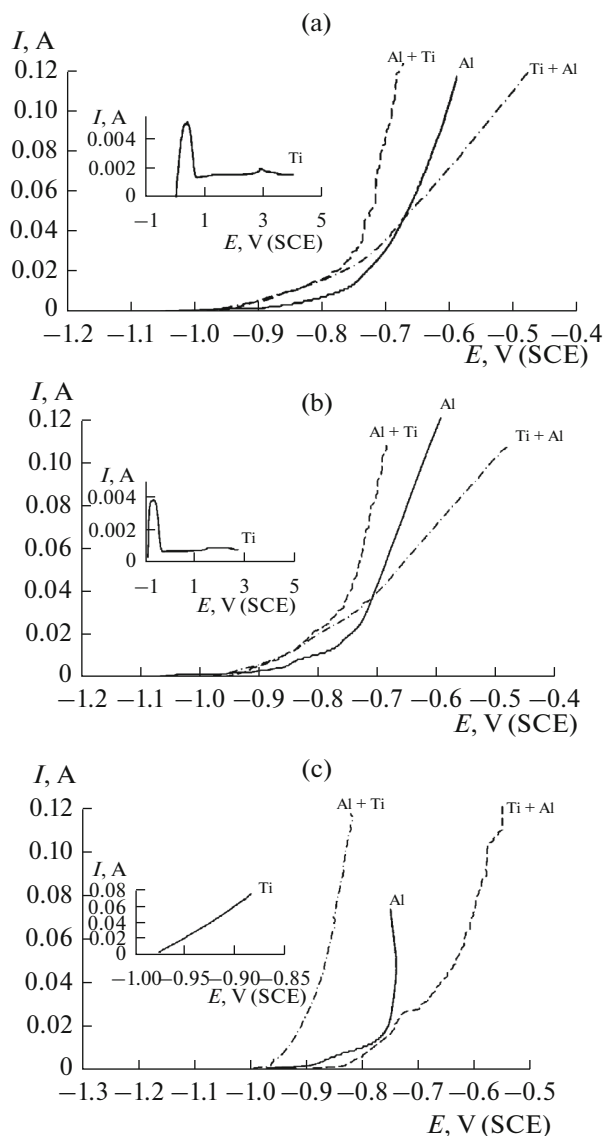


Fig. 1. Anodic polarization curves of aluminum and titanium upon ratio of surfaces $S(\text{Al}) : S(\text{Ti}) = 1 : 1$ in 1.0 M NaCl solution with HF as an additive: (a) 0.05, (b) 0.1, and (c) 0.5 mol/L. Designations: Al and Ti are individual metals (aluminum and titanium) that are not in contact with each other, Al + Ti is aluminum (as part of a hybrid electrode) in contact with titanium, and Ti + Al is titanium (as part of a hybrid electrode) in contact with aluminum.

Anode process depends on electrolyte composition (corrosive medium), composition and properties of metals, their electrode potentials, and ratio of the contacting surfaces.

No active anodic dissolution occurs and metal potential shifts toward positive values (Figs. 1a and 1b) during anodic polarization of titanium in solutions containing hydrofluoric acid as an additive upon its concentrations from 0.01 to 0.1 mol/L. The polarization curves of an individual titanium anode contain an extended passivity region, whereas values of anode

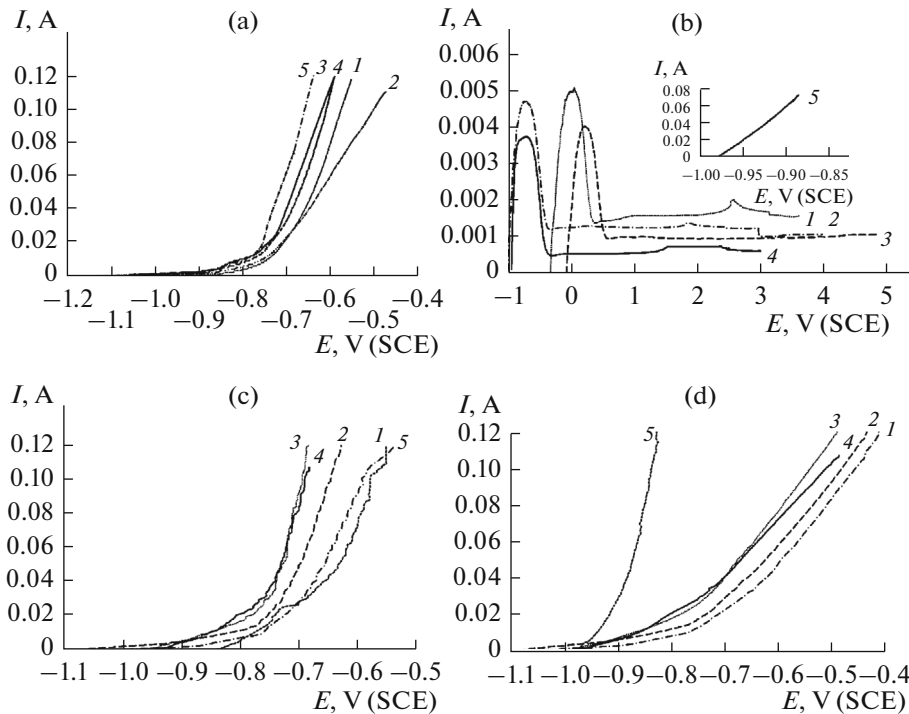


Fig. 2. Anodic polarization curves of (a) Al, (b) Ti (that are not in contact with each other), (c) Al + Ti, and (d) Ti + Al components of a hybrid electrode upon a ratio of surfaces $S(\text{Al}) : S(\text{Ti}) = 1 : 1$ in 1.0 M NaCl solution with HF as an additive: (1) 0.01, (2) 0.025, (3) 0.05, (4) 0.1, and (5) 0.5 mol/L.

dissolution currents are small, of the order of 5 mA. Titanium in the hybrid electrode, however, begins to dissolve as intensively as aluminum (Figs. 1a, 1b). When amount of HF increases up to 0.5 mol/L, the behavior of anodic polarization changes: the rate of anodic dissolution of titanium, being individual and involved into a hybrid electrode, increases (Fig. 1c). Under these conditions, titanium actively interacts with fluoride ions to form highly soluble fluoride complexes, thereby facilitating ionization [21].

Figure 2 shows the effect of hydrofluoric-acid concentration on the polarization characteristics of the electrodes under study. When fluoride concentration in the electrolyte increases, the metal potential shifts by approximately 100–150 mV toward more negative values during polarization of aluminum, which does not contact with titanium (Fig. 2a). An increase in HF concentration leads to a shift of the current peak of the polarization curve by approximately 1000 mV toward more negative potential values (Fig. 2b). All this indicates that a titanium surface is activated, accompanied by its dissolution.

Titanium in the hybrid electrode under anodic polarization conditions is also sensitive to an increase in fluoride concentration (Fig. 2d): the electrode potential at the beginning of intensive dissolution shifts toward negative values, about 200–300 mV. The polarization parameters of a hybrid electrode depend also on area ratios of the working surfaces of its com-

ponents (Fig. 3). It is seen that, when the aluminum working surface increases relative to titanium, the active dissolution potential of the electrodes making up the contact pair shifts toward more negative values. This pattern remains unchanged in all the solutions studied.

It is known that, when metals are in contact with different electrochemical potentials, the rate of their corrosion can vary significantly. When current appears in such a system, the potentials for anode and cathode change become opposite: the cathode potential appears to be more electronegative, whereas the anode one is more electropositive.

Figures 4 and 5 show typical chronopotentiograms of the working electrodes. It can be seen that the potentials of the contacting metals are as close as possible upon zero or low polarization (Fig. 4) and differ by a value that is apparently equivalent to the difference of galvanic potentials for these metals. When polarization is higher (Fig. 5), the potentials of the contacting metals, however, differ a great deal.

An increase in the current of anodic polarization leads to more intense dissolution (ionization) processes, which begin to predominate over those phenomena, which occur during current-free contact of metals to change the potential/time relationship. An increase in anode current (Fig. 6) leads to a displacement of the potential of the contacting metals toward more positive values.

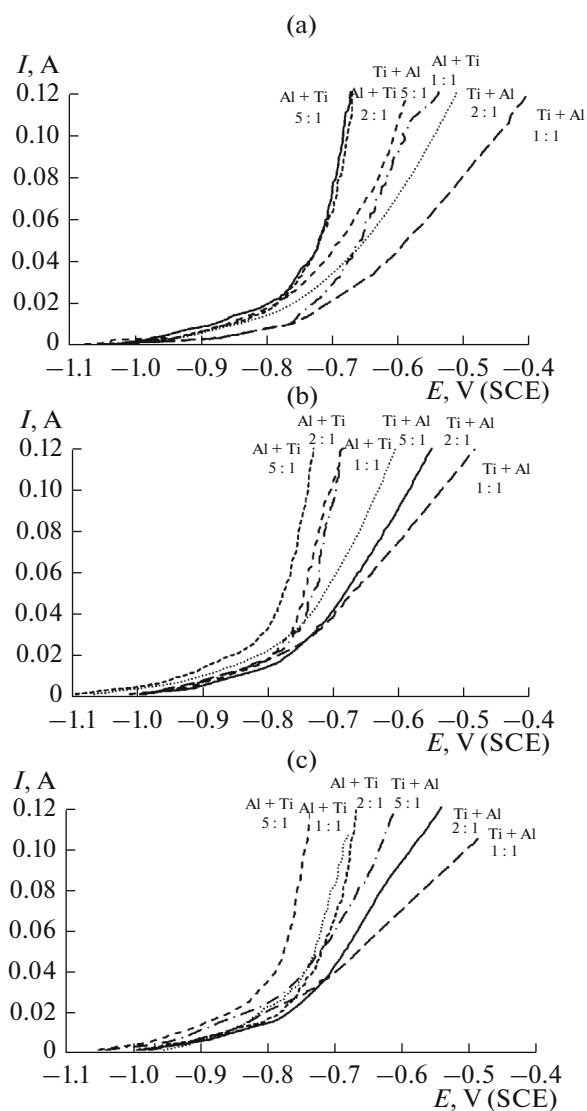


Fig. 3. Anodic polarization curves of a hybrid electrode upon different surface ratios $S(\text{Al}) : S(\text{Ti})$ in 1.0 M NaCl solution with HF as an additive: (a) 0.01, (b) 0.05, and (c) 0.1 mol/L. Designations: Al + Ti is aluminum (as part of a hybrid electrode) in contact with titanium and Ti + Al is titanium (as part of a hybrid electrode) in contact with aluminum.

The oxidation rates for both separate and contacting metals were found through the gravimetric method so as to study the joint anodic dissolution process of aluminum and titanium. The rate of metal oxidation (V_{ox}) decreased in its weight per time (1 h), referring to the working surface of electrode. Table 1 and Fig. 7 show the data obtained. It is seen that there is an effect of current applied and of the ratio of the area of the working surfaces for aluminum and titanium in the hybrid electrode on the intensity of anodic dissolution of metals in the corresponding halide solutions.

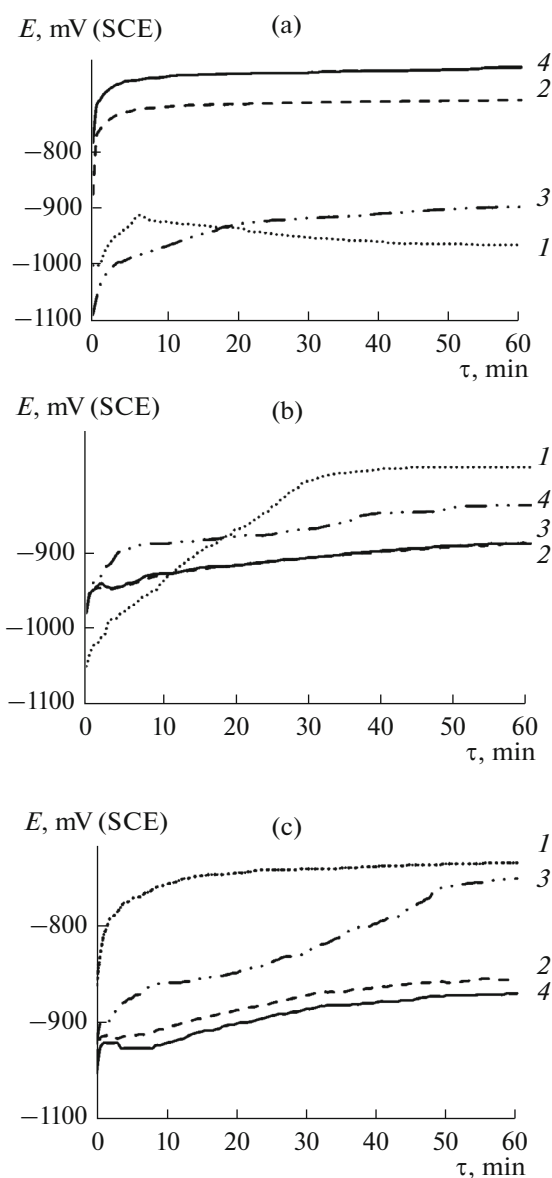


Fig. 4. Change in electrode potential in 1.0 M NaCl solution with HF as additive: (a) 0.05 and (b, c) 0.1 M under (a, b) open-circuit conditions and (c) anode current strength of 10 mA: (1) Al (individual metal), (2) Al (in hybrid electrode), (3) Ti (individual metal), and (4) Ti (as part of a hybrid electrode). The ratio of surfaces $S(\text{Al}) : S(\text{Ti})$ is 1 : 1.

The experimental data indicate (Table 1) that titanium incorporated into a hybrid electrode (upon equal working surfaces of aluminum and titanium) and as individual metal dissolves mainly without polarization and upon low anode current values (≤ 10 mA) in the 1.0 M NaCl electrolyte with HF (≥ 0.1 mol/L) as an additive (Fig. 7a). Considering that the solubility of titanium in hydrofluoric acid is high [22], most of the loss in weight is due to its chemical dissolution: oxidation rate, for example, is no more than 3 mg/cm² h, when anode current is 50 mA (Fig. 7a). At the same

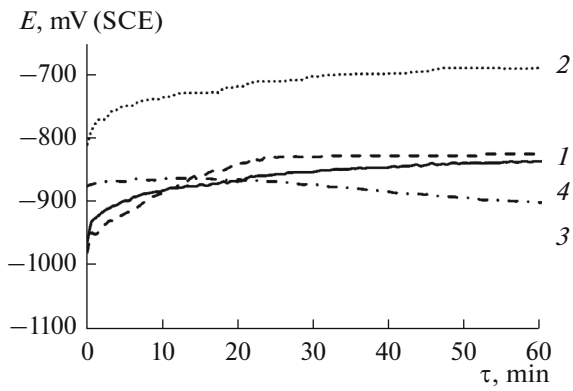


Fig. 5. Change in electrode potential in 1.0 M NaCl/0.5 M HF solution upon anode current of 50 mA: (1) Al (individual metal), (2) Al (in the hybrid electrode), (3) Ti (individual metal), and (4) Ti (as part of a hybrid electrode). The ratio of surfaces $S(\text{Al}) : S(\text{Ti})$ is 1 : 1.

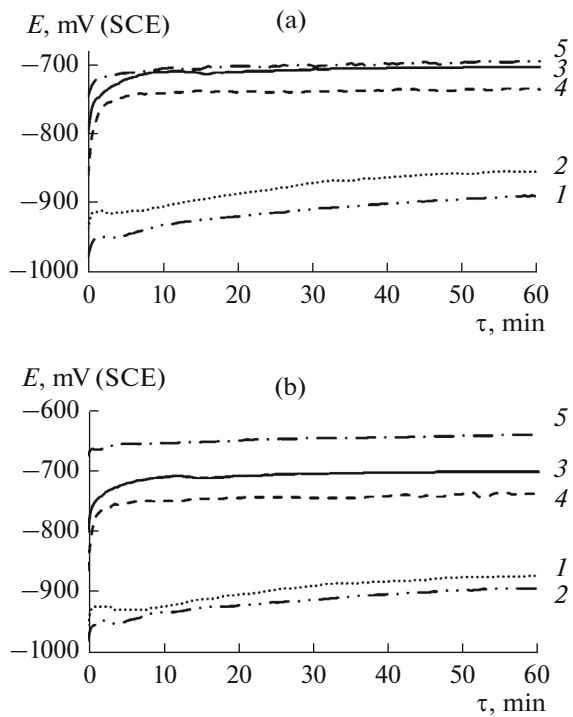


Fig. 6. Change in potential of (a) aluminum in contact with titanium and (b) titanium in contact with aluminum in 1.0 M NaCl/0.1 M HF solution upon anode current value (I) 0, (2) 10, (3) 25, (4) 50, and (5) 75 mA. The ratio of surfaces $S(\text{Al}) : S(\text{Ti})$ is 1 : 1.

time, aluminum involved into the hybrid electrode dissolves quite intensely in the 1.0 M NaCl/0.1 M HF electrolyte, although to a lesser extent than the separate metal (Fig. 7a). When the current value is 75 mA, the dissolution rate of the aluminum component of the hybrid electrode exceeds that of titanium by about seven times (Table 1, Fig. 7a).

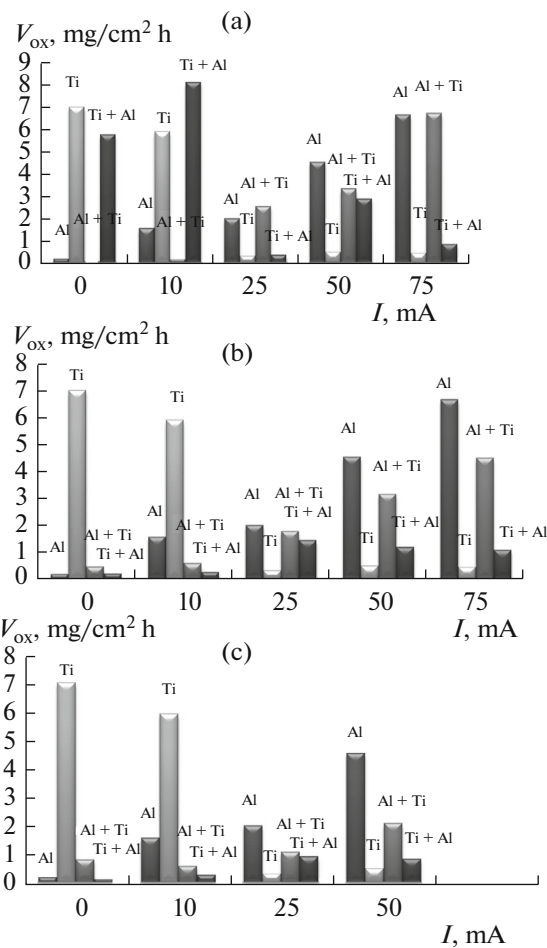


Fig. 7. Oxidation rate for individual and contacting metals in a hybrid electrode under anode galvanostatic polarization in 1.0 M NaCl/0.1 M HF solution upon different ratios of the aluminum surface to that of titanium: (a) 1 : 1, (b) 2 : 1, and (c) 5 : 1.

When the proportion of hydrofluoric acid increases in the electrolyte, titanium dissolves preferentially, whereas the dissolution rate of aluminum decreases sharply (Table 1).

When the surface of the aluminum component in the hybrid electrode doubles relative to the titanium one, the situation remains unchanged (Table 1, Fig. 7b): the dissolution process of titanium predominates, whereas, when the anodic polarization is ≥ 50 mA, aluminum dissolves three to five times more intensely than does titanium. When the surface of the aluminum is five times greater than that of the titanium, the dissolution rates for both components of the hybrid electrode become equal and the decrease in weights for both titanium and aluminum is comparable (Fig. 7c).

Thus, intensity of anodic dissolution of the constituents in the hybrid electrode in the electrolyte varies depending on the ratio of the working surfaces of the contact pairs in the hybrid electrode, current applied, and concentration of hydrofluoric acid. The condi-

Table 1. Rate of anodic oxidation for aluminum and titanium

Solution composition	S(Al) : S(Ti)	Electrode*	Oxidation rate of metal, mg/cm ² h under different current values, mA				
			0	10	25	50	75
1.0 M NaCl + 0.01 M HF	1 : 1	Al	0.05	0.87	2.05	3.30	1.67
		Ti	0.90	0.41	0.16	0.24	0.24
		Al + Ti	0.15	0.83	2.13	4.33	5.63
		Ti + Al	0.12	0.24	0.16	0.90	0.07
	2 : 1	Al + Ti	0.12	0.49	1.37	2.74	4.00
		Ti + Al	0.07	0.2	0.03	0.27	0.08
	5 : 1	Al + Ti	0.26	0.57	1.57	2.97	
		Ti + Al	0.09	0.11	0.71	0.38	
1.0 M NaCl + 0.05 M HF	1 : 1	Al	0.21	1.04	2.44	4.20	6.20
		Ti	0.76	0.36	0.26	0.18	0.32
		Al + Ti	0.83	0.76	1.68	4.12	3.96
		Ti + Al	0.41	0.57	0.84	0.63	2.09
	2 : 1	Al + Ti	1.31	0.70	1.53	3.13	2.19
		Ti + Al	0.37	0.65	1.57	0.50	0.19
	5 : 1	Al + Ti	0.72	0.95	1.40	2.21	
		Ti + Al	0.58	0.33	1.13	0.45	
1.0 M NaCl + 0.1 M HF	1 : 1	Al	0.19	1.57	2.01	4.55	6.68
		Ti	7.02	5.92	0.32	0.50	0.45
		Al + Ti	0.08	0.16	2.56	3.35	6.74
		Ti + Al	5.78	8.14	0.37	2.89	0.85
	2 : 1	Al + Ti	0.47	0.59	1.79	3.17	4.51
		Ti + Al	0.21	0.27	1.45	1.19	1.09
	5 : 1	Al + Ti	0.82	0.59	1.08	2.09	
		Ti + Al	0.12	0.27	0.92	0.84	
1.0 M NaCl + 0.5 M HF	1 : 1	Al		0.02		0.05	
		Ti		53.14		51.26	
		Al + Ti		0.275		0.87	
		Ti + Al		1.08		1.38	

* Al and Ti are individual metals (aluminum and titanium), which are not in contact with each other, Al + Ti is aluminum in contact with titanium, and Ti + Al is titanium in contact with aluminum.

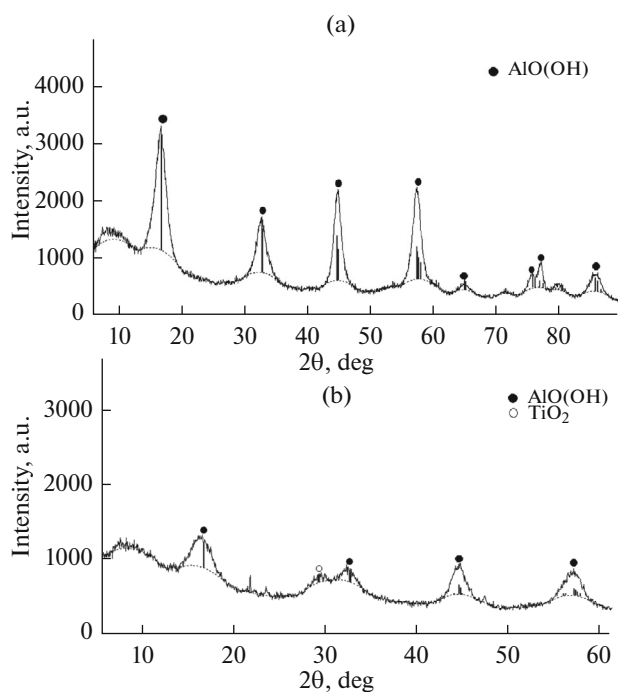


Fig. 8. X-ray diffraction pattern of metal oxidation product of a hybrid electrode treated thermally under 80°C and prepared via electrolysis in 1.0 M NaCl with (a) 0.01 and (b) 0.05 M HF. The ratio of surfaces S(Al) : S(Ti) is 2 : 1.

tions for dissolution of a hybrid electrode can be selected by varying those for anodic polarization in a sodium chloride solution and, depending on the task at hand, achieving either dissolution with similar rates of both electrode components or predominant dissolution of a particular metal. As a result, it is possible to control the anodic dissolution process of a hybrid electrode.

The metal oxidation reaction to form a precipitate, for example, proceeds only on aluminum until its complete dissolution (47 min), and the weight loss of titanium component in the hybrid electrode is zero

during electrochemical synthesis in 1.0 mol/L sodium chloride aqueous solution with 0.01 mol/L hydrofluoric acid as an additive upon the ratio of the working area of the contacting metals S(Al) : S(Ti) = 2 : 1 and a current density of 100 mA/cm². A white powder formed after filtering and drying the precipitate at 80°.

X-ray phase analysis of the product obtained under these conditions (Fig. 8a) indicates that aluminum hydroxide with a boehmite structure forms (100%).

In addition, the changes in the surfaces of aluminum and titanium components in the hybrid electrode have different behaviors. Electron-microscopic data of the hybrid electrode show that there are numerous points on the aluminum surface, that have a well-defined spherical shape with a diameter of ~100–150 μm (Fig. 9). The titanium surface after anodic polarization remains smooth and has no corrosion lesions; only small foci are observed on it.

Amount of HF in the electrolyte and electrolysis time in further experiments were increased up to 0.05 mol/L and 80 min, respectively. In this case, complete dissolution of aluminum occurs within 45 min.

X-ray diffraction data (Fig. 8b) indicate that aluminum hydroxide with a boehmite γ -AlOOH structure (73.6%) and titanium oxide with an anatase structure (26.4%) form during drying the electrolysis product at 80°C. Table 2 outlines X-ray phase analysis results depending on electrolysis conditions (solution composition, electrical regime, and process duration).

Through pores with a diameter from 30 to 120 μm form on the aluminum surface after electrolysis in 1.0 M NaCl/0.05 M HF solution. The titanium surface is destroyed mainly along the edges of the electrode to form damaged areas of various shapes, mainly rounded oblong ones, the sizes of which are within ~3–6 μm (Fig. 10).

It is known that the phase composition of a precipitate is directly due to crystallization processes that occur immediately during precipitation and maturation in a mother liquor [3, 14]. Al³⁺ and Ti⁴⁺ ions

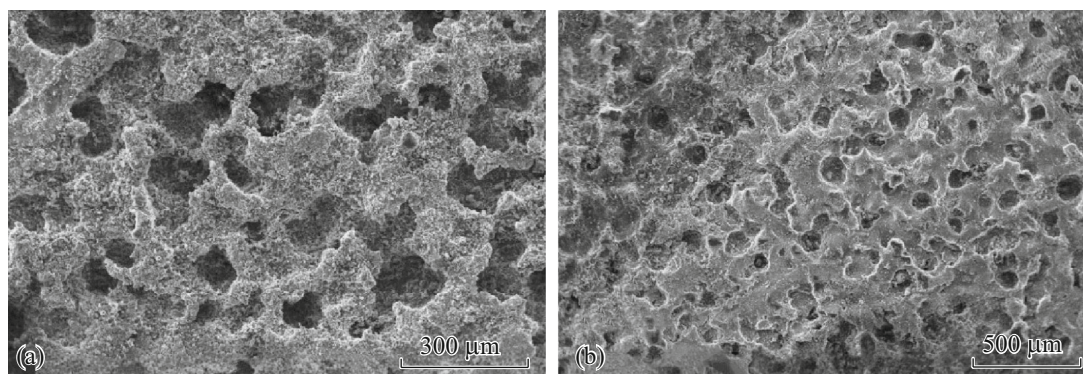


Fig. 9. Microphotographs of aluminum surface in a hybrid electrode after electrolysis ($j = 100$ mA/cm², $\tau = 47$ min, and S(Al) : S(Ti) = 2 : 1) in 1.0 M NaCl + 0.01 M HF solution.

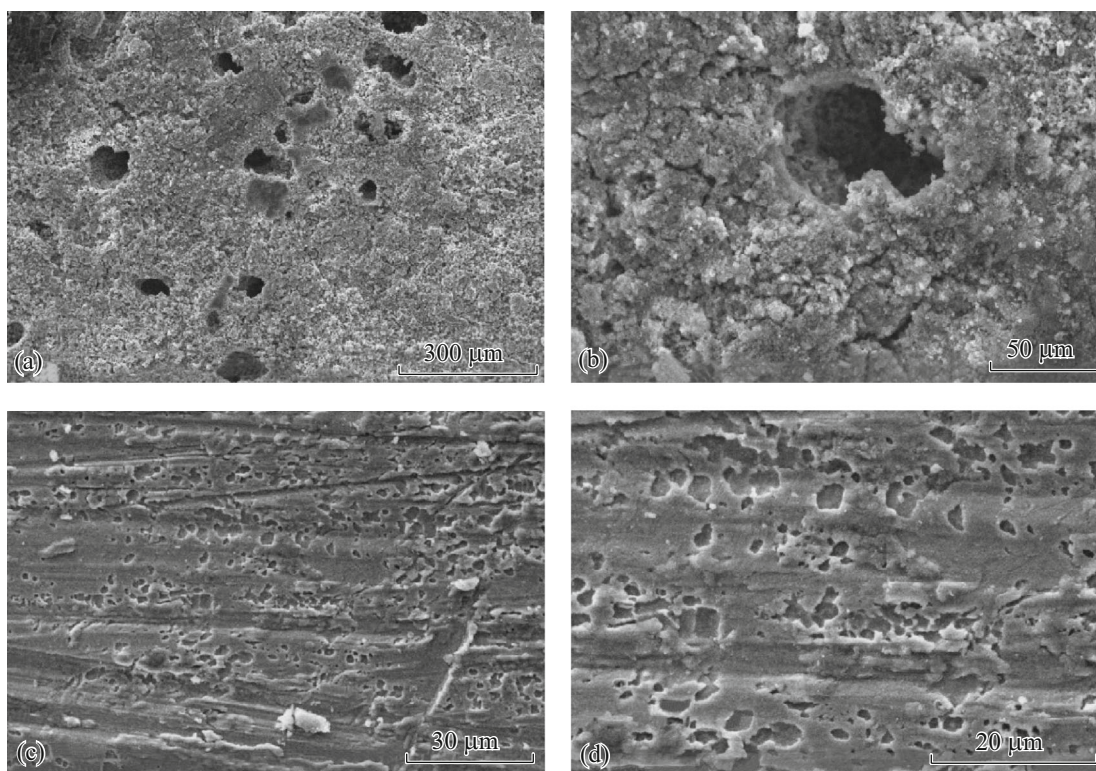


Fig. 10. Microphotographs of (a, b) aluminum and (c, d) titanium surfaces in a hybrid electrode after electrolysis ($j = 100 \text{ mA/cm}^2$, $\tau = 80 \text{ min}$, and $S(\text{Al}) : S(\text{Ti}) = 2 : 1$) in 1.0 M NaCl/0.05 M HF solution.

undergo main hydrolysis stages under different pH values of the solutions. The most developed particle structure forms during precipitation under low pH values; in addition, there is a large number of mesopores and micropores [14, 19].

The microimage of the precipitate obtained (Fig. 11) indicates that it has a developed surface and submicron particles. The sample under study is obviously a set of aggregates of different sizes and shapes. The local elemental analysis of the sample site indicated in the photomicrograph provided the following chemical composition: F, Na, Al, Si, Cl, Ti, and O, the amounts of which were 6.99, 0.97, 38.68, 0.50, 1.11, 9.85, and 41.89 wt %, respectively. This confirms indirectly the X-ray phase analysis data reported previously.

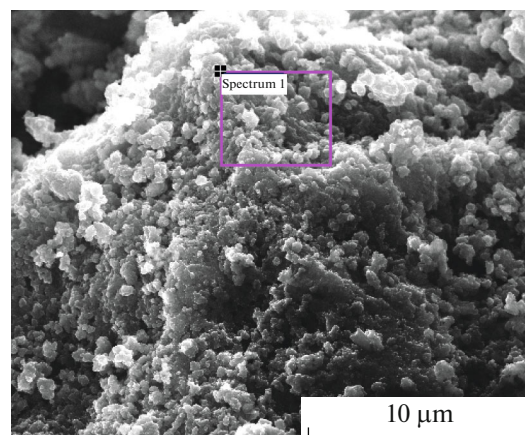


Fig. 11. Microimage of the “boehmite–anatase” system synthesized by the electrochemical method ($j = 100 \text{ mA/cm}^2$, $\tau = 80 \text{ min}$, and $S(\text{Al}) : S(\text{Ti}) = 2 : 1$) in 1.0 M NaCl/0.05 M HF solution.

Table 2. Phase composition of the electrolysis products ($j_a = 100 \text{ mA/cm}^2$, and $S(\text{Al}) : S(\text{Ti}) = 2 : 1$)

Electrolysis conditions		Amount of phases, wt %					
solution composition	time, min	boehmite $\gamma\text{-AlOOH}$	CSR, Å	bayerite $\alpha\text{-Al(OH)}_3$	CSR, Å	anatase TiO_2	CSR, Å
1 M NaCl + 0.01 M HF	47	100	47.0	—	—	—	—
1 M NaCl + 0.025 M HF	60	99.3	40.6	0.07	658.3	—	—
1 M NaCl + 0.05 M HF	80	73.6	40.1	—	—	26.4	64.1

CONCLUSIONS

(1) The anodic behavior of a hybrid titanium–aluminum electrode in solutions containing chloride and fluoride ions was studied for the first time.

(2) The possibility of controlling the rate of anodic reactions and composition of anodic oxidation products for aluminum and titanium components in a hybrid electrode via changing the anode current density and ratio of the working surfaces of metals has been experimentally confirmed.

(3) An increase in hydrofluoric-acid concentration in electrolyte up to 0.05 mol/L has a slight effect on the rate of titanium anodic dissolution.

(4) A highly dispersive system consisting of boehmite and anatase forms during anodic dissolution of a hybrid titanium–aluminum electrode ($S(\text{Al}) : S(\text{Ti}) = 2 : 1$, $j = 100 \text{ mA/cm}^2$, and $\tau = 80 \text{ min}$) in 1.0 M NaCl/0.05 M HF electrolyte.

ACKNOWLEDGMENTS

This work was performed on equipment of the Nanomaterials and Nanotechnologies Center for Collective Use for obtaining and studying the nanoparticles of metals, metal oxides, and polymers within state assignment no. 4.1584.2014/k (2014–2016).

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Translated by A. Tulyabaev