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Abstract: Aims: Anodic polarization behavior of a combined iron-titanium electrode (two metals in electrical contact with each other) in aqueous solutions containing halide ions (F⁻ and Cl⁻) was studied.

Methods: The joint anodic dissolution of titanium and iron with subsequent thermal treatment makes it possible to obtain precursors of a highly dispersed mixed oxide system Fe₂O₃-TiO₂. The phase and elemental composition and structural characteristics of obtained products were examined using X-ray diffraction and scanning electron microscopy. It has been experimentally confirmed that via changing the anode current density, hydrofluoric acid concentration in electrolyte and ratio of the working surface area of contacting metals, it is possible to effectively control the rate of anodic reactions and phase composition and morphology of anodic oxidation products for iron and titanium components in a combined electrode.

Results: The main results of this study are as follows: Electrochemical method for the synthesis of complex dispersed oxide system Fe₂O₃-TiO₂ based on joint anodic oxidation of contacting metals in aqueous media was suggested. Relationships between parameters of the electrochemical process and characteristics of the synthesized oxide system were revealed.

Conclusion: By varying the parameters of the electrolysis process, it is possible to prepare complex oxhydroxides with different ratios of iron and titanium, which makes it possible to synthesize precursors of iron titanates of preset composition and structure.

Keywords: Iron-titanium electrode, anodic dissolution, precursors, Fe₂O₃-TiO₂ oxide system, hydrofluoric acid, X-ray.

1. INTRODUCTION

Recently, solid solutions of such minerals as ilmenite-hematite (Fe₂₃Ti₂O₇), ulvospinel-magnetite (Fe₃₋ₓTiₓO₄), pseudobrookite (Feₓ₋₂₊₂TiₓO₅), their electronic structure, phase composition, physicochemical properties are the subject of intensive and intent study [1-4]. First of all, this interest is due to the fact that some solid solutions of the Fe-Ti-O system are potentially spintronic materials because such compositions (e.g. (FeTiO₃)₁₋ₓ(Fe₂O₃)ₓ in the range 0.15<x<0.5) are known to be semiconducting and also magnetic [2, 3].

It is noted [5-7], that in contrast to pure TiO₂ phases, with active absorption in the UV region, Fe₂O₃-TiO₂-based nanomaterials (iron-doped TiO₂ or mixed Ti/Fe oxides) are photocatalytic in the presence of visible light. Besides, such a photo-catalyst can be easily and unproblematic removed from the liquid or gas phase through an applied magnetic field.

The combination of dielectric and magnetic properties makes it possible to use titanium oxide/iron composites as microwave absorption material, for example, in shielding against electromagnetic radiation in military, commercial, and scientific electronic devices [8].

Composites based on nanostructured mixed iron-titanium oxides are among the most interesting and promising anodic materials for use in Li-ion batteries [9, 10]. Besides, iron-titanium oxides materials are in great demand in various practical applications, particularly as gas sensors, solar cells, supercapacitors, and efficient sorbents [11-14].

There are various methods for the synthesis of complex oxide system Fe₂O₃-TiO₂, such as mechanochemical methods [9], but in most cases mainly chemical methods are used [5, 6, 8, 15]. Thus, in a study [15], spherical micronsized particles of binary mixtures of goethite α-FeOOH and anatase phase of TiO₂ were synthesized using homogeneous precipitation from aqueous solution using urea as the precipitation agent in the presence of corresponding metal sulfates.

Besides, a number of electrochemical methods have been successfully realized for the synthesis of such complex oxide systems [7, 12, 14]. For instance, another study [7] reported on the synthesis of rutile phase titania nanofibres by electrospinning and its doping with ferric oxide via precipitation method. It was found [7] that the obtained samples of titania nanofibres doped with ferric oxide demonstrated the highest photocatalytic activity in the process of dye degradation, in
particular Congo red, as compared to commercially available titanium nanoparticles and pristine nanofibers. A simple electrospinning technique was used to obtain novel Fe-doped amorphous TiO₂/C nanofibres for supercapacitors application [14].

At present, the actual task is to develop environmentally safe, simple and accessible ways for obtaining metal oxides in the form of ultradispersed particles with high product yield and also with reproduced and controlled properties. In this regard, electrochemical synthesis, based on metals anodic ionization and subsequent hydration, is a promising method for the production of metal oxides. It provides the possibility to regulate process parameters (composition and concentration of electrolyte, current, voltage, etc.) and allows to obtain dispersed products of high purity with definite predetermined characteristics (morphology, shape, a certain range of sizes, element and phase composition).

Electrochemical method for the preparation of ultra- and nanodispersed metal oxides was proposed for various metals so far [16-20], including titanium dioxide [16-18] and iron oxide [19]. However, there are no data on the synthesis of mixed Fe₂O₃-TiO₂ oxide systems, based on electrochemical oxidation. Previously [21, 22] we have synthesized mixed Al₂O₃-Fe₂O₃ nanosized system using joint anodic dissolution of aluminum and iron and subsequent heat treatment of the precipitate. In these reports [21, 22], we have established the relationship between electrolysis parameters and characteristics of the synthesized oxide system.

This study mainly focused on the electrochemical behavior of a combined iron-titanium electrode under conditions of anodic polarization in aqueous solutions, containing sodium chloride and hydrofluoric acid. We aimed to investigate the influence of process conditions (solution composition, current density, the ratio of the working surfaces of the composite electrode components) on the intensity of its anodic dissolution and characteristics of the synthesized oxide system Fe₂O₃-TiO₂.

2. EXPERIMENTAL

The test electrode for electrochemical measurements was a combined electrode consisting of two metals in contact with each other – technical iron (99.19% purity) and titanium (purity not less than 98.33%). The components of the combined electrode (plates of iron and titanium) were located close to each other, along the sidewall of a rectangular cell. Exposed apparent area of the test electrode (Fe-Ti combined electrode) was 14.7 cm². The ratio of working surfaces of iron and titanium was changed, S(Fe):S(Ti) = 1:1; 2:1; 5:1. The test electrode was pre-treated by mechanical polishing using diamond paste, chemical degreasing with ethanol, and rinsing with double-distilled water.

The electrochemical study was performed in a four-electrode cell with a steel (X10CrNiTi18-10) auxiliary electrode (cathode), two commercial Ag/AgCl reference electrodes (for simultaneous measuring of Fe and Ti potential) and the working electrode (Fe-Ti combined anode). The distance between cathode and anode was 150 mm. The interval of current density change was 0.1 mA/cm², potential delay was 30 s. Polarization measurements were performed in natural aerated aqueous solutions containing sodium chloride and hydrofluoric acid under standard conditions (101.3 kPa, 298 K). Polarization of the working electrode was provided on the electrochemical station model Zive SP2 (WonATech). Simultaneous measurement of iron and titanium potential was provided by means of computer-controlled potentiostat-galvanostats model P-301M (Elins).

Synthesis of precursors of binary iron and titanium oxides-hydroxides was carried out via electrolysis in coaxial reactor (free diaphragm) of 500 ml (electrolyte volume 300 ml) by DC power supply (MATRIX, model MPS-7101). The central electrode (cathode) in the coaxial reactor was a steel wire X10CrNiTi18-10 with 1.5 mm diameter. Combined electrode (soluble anode) consisted of alternating and rigidly bonded iron and titanium plates, soldered to the copper wire. The working surface area of the anode was two or more orders (≥100) higher than the surface of the cathode (from 36 to 54 cm²). The distance between anode and cathode was ~40 mm, current density equaled to 100-200 mA/cm², duration of electrolysis amounted to 60-150 min. The ratio of working surfaces of iron and titanium was: S(Fe):S(Ti) = 2:1 and 5:1. All the experiments were performed at room temperature (~25°C) in 1.0 M NaCl (of analytical grade) aqueous solution with the addition of hydrofluoric acid (0.1; 0.5; 1.0 mol/l).

The obtained precipitate for crystallization was kept in a matrix solution for 24 h, after which it was filtered and dried at 80°C for 24 h in ambient atmosphere. Afterward, the sample was calcined at 1100°C (for 2 h) in order to obtain oxides.

Phase analysis of the synthesized powders was conducted by X-ray diffraction standard technique with a D2 PHASER diffractometer using monochromated Cu Kα radiation (λ=1,78897 Å) operating at 30 kV voltage and 10 mA current. Patterns of Fe₂O₃–TiO₂ powders were recorded from 3° to 130° (in 2θ) with a 0,05° step width and a 1 s counting time per step. The calculation of interplanar distances of diffraction reflections was automatically carried out using a DIFFRACT. EVA software program package.

The morphology of Fe₂O₃–TiO₂ powders was investigated by an EVEX Mini SEM SX-3000 scanning electron microscope (SEM), with simultaneously performing the elemental analysis (15 kV, 10 µA). The local chemical composition, microstructure parameters and size of particles were studied using an Auriga™ CrossBeam® Zeiss high-resolution auto emission electron microscope with energy dispersive spectrometry INCA X-MAX (resolution 127 eV) operated at an acceleration voltage of 20 kV. To increase the conductivity of the samples, they were gold-palladium (60:40) coated (15 nm) using Quorum Q150T ES.

3. RESULTS AND DISCUSSION

In order to obtain a mixed dispersed oxide system Fe₂O₃-TiO₂, the primary and important research task was to determine the optimal conditions for anode-anion activation and ionization of the combined electrode. Due to the fact that titanium is easily passivated during anodic polarization in aggressive media, including solutions of chlorides and many oxidants, there is a serious obstacle to its use as a soluble anode.
According to known technologies [16-18], the electrochemical synthesis of porous titanium oxide is possible to implement in fluoride polar organic electrolytes or in aqueous solutions of hydrofluoric acid at high oxidation potentials. In our previous studies [21, 22], the process of joint iron and aluminum anodic dissolution has been carried out in an aqueous solution of 0.1 M sodium chloride by means of direct or alternating current of an industrial frequency of 50 Hz.

In the present study, it has been previously established that sodium chloride solution (1.0 mol/l) is the most acceptable and universal electrolyte for performing joint iron and titanium anodic dissolution at a relatively high rate of oxidation of both metals. In order to activate the surface of titanium, hydrofluoric acid (in the concentration range 0.01 to 1.0 mol/l) was added to the initial solution (1.0 M NaCl). In the presence of hydrofluoric acid, titanium reacts actively with fluorine ions to form complex ions of fluoride compounds [23], thereby facilitating its dissolution.

In the course of preliminary studies, it has been shown that anodic oxidation rate of a metal ($V_{an}$) is determined by the magnitude of applied current, the ratio of working surfaces of iron and titanium in the combined electrode, and the concentration of acid (Fig. 1). For the oxidation rate ($V_{an}$), the weight loss of the corresponding electrode per unit of time (1 h), related to the unit of its working surface, has been taken.

According to obtained data, titanium component of the combined electrode (with equal surfaces of iron and titanium) dissolves very little in sodium chloride solutions with the addition of HF < 0.5 mol/l and, mainly, in the absence of polarization (Fig. 1a), which is due to its high chemical solubility in hydrofluoric acid [23]. At the same time, under identical conditions, iron component of the combined electrode, as well as iron electrode (individual, non-contacting metal), dissolves quite intensively at the values of anodic current ≥ 25 mA (Fig. 1a). The oxidation rate of iron component (in contact with titanium; $S(Fe)$:$S(Ti)$ = 1:1) in the 1.0 M NaCl + 0.1 M HF solution increases approximately 2-fold with an increase of current from 20-25 to 70-75 mA. When the current reaches 70-75 mA, the intensity of its dissolution exceeds the dissolution of the titanium component by an order of magnitude or more (Fig. 1a).

The preferential dissolution of titanium is observed in solutions with an increased concentration of hydrofluoric acid (≥ 0.5 mol/l), while the dissolution rate of iron falls

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**Fig. (1).** Oxidation rate of iron and titanium (individual metals and contacting metals as components of the combined electrode) under anodic polarization in (a, c) 1.0 M NaCl + 0.1 M HF and (b) 1.0 M NaCl + 1.0 M HF solutions at the surface ratio $S(Fe)$:$S(Ti)$ (a, b) 1:1 and (c) 5:1. Key: 1 – Fe (electrode); 2 – Fe (component of the combined electrode); 3 – Ti (electrode); 4 – Ti (component of the combined electrode).
sharpely (Fig. 1b). In this case, on the contrary, the oxidation rate of titanium component exceeds the dissolution rate of iron component by more than two orders of magnitude.

In the case, where the surface of iron component is more than twice the surface of titanium component \((S_{(Fe)}:S_{(Ti)} = 2:1)\), the picture does not change in principle – the ionization of iron component prevails, and under conditions of considerable anodic polarization \((\geq 50\, \text{mA})\) in the 1.0 M NaCl + 0.1 M HF solution it dissolves on average 5-10 times more intensively than titanium component.

However, in the case of a more significant predominance of the surface of iron, for instance, \(S_{(Fe)}:S_{(Ti)} = 5:1\) (Fig. 1c), the values of the oxidation rate of contacting metals are aligned and become comparable.

Thus, it is possible to control the rate of reactions of iron and titanium anodic dissolution in the chloride-fluoride electrolyte by varying polarizing current, concentration of HF, and ratio of working surfaces of metal components. In addition, depending on the task, it is possible to achieve either predominant dissolution of iron or titanium, or relatively uniform dissolution, with close rates, of both electrode components.

Figs (2 and 3) show typical "potential-time" curves of working electrodes. It may be seen that at zero (Fig. 2a) or low polarization (Fig. 2b) potentials of the contacting metals are maximally close, in comparison with the potentials of non-contacting metals, but do not coincide and differ by an amount that is equivalent to the galvanic potentials difference of these of metals. Electrode potentials change with time slightly, and this indicates the stability of the process.

The HF concentration, as well as the value of anodic current influences the "potential-time" dependence (Fig. 3). For example, the increase of anodic current shifts the potential of contacting metals to a region of more positive values, by 200-400 mV. There is a more intensive course of dissolution (ionization) processes in this case, which begins to predominate over the phenomena that occur during the contact of metals, and the character of the "potential-time" dependence varies. It is also observed (Figs. 3a and 3b) that the increase of acid content (in the absence of polarization) promotes a shift in the potentials of contacting metals to a region of more negative values, on average by 300-400 mV. However, this shift becomes less significant (50-100 mV) with the increase of polarizing current (Figs. 3c and 3d).

Characteristic curves of anodic electrode polarization in chloride solutions with the addition of HF (0.1 and 0.5 M) are presented in Figs. (4 and 5), respectively. The polarization curves of titanium electrode (curves 3 in Figs. 4 and 5) have a typical form – an active zone with low anodic dissolution currents (value of the order of 5 mA), followed by an extended passivity region, caused by the formation of a passive film with a decrease in current by 5-10 times. However, the polarization curve of titanium component (in contact with iron) has a different form (curves 4 in Figs. 4 and 5). It may be seen that active dissolution of titanium component in the 1.0 M NaCl + 0.1 M HF solution begins in the range of potentials from (-0.10) to 0.10 V (curves 4 in Fig. 4) and its dissolution rate increases by more than an order of magnitude in comparison with titanium electrode (individual metal, without contact). According to obtained data, in all cases (Figs. 4a-4c) the dissolution rate of iron component is higher than the dissolution rate of titanium component. However, when the surface area of iron is more than titanium surface five times, these rates become comparable (curves 2 and 4 in Fig. 4c).

The addition of more hydrofluoric acid (0.5 mol/l) to the solution influences the kinetics of anodic oxidation of both metallic components (Figs. 5a-5c). It can be seen more distinct and extended area corresponding to the passivity region on the polarization curves of iron, especially in the case when the surface of iron component is larger (curves 2 in Figs. 5b and 5c). In contrast to this, an increase in the ratio of working surfaces of iron and titanium leads to a decrease in the extent of the passivity region on the polarization curves of titanium; in addition, it becomes less pronounced. It should be noted that under anodic polarization the increase of HF concentration promotes a shift in activation potential of titanium component to a region of more negative values on average by 300-400 mV (Figs. 5a-5c). We can conclude

Fig. (3). Change in the potential of (a, c) iron in contact with titanium and (b, d) titanium in contact with iron (a, b) at open circuit and (c, d) under galvanostatic anodic polarization for constant current density, $j = 8.3 \text{ mA/cm}^2$ in 1.0 M NaCl + n M HF, where n, mol/l: 1 – 0.05; 2 – 0.1; 3 - 0.5.

Fig. (4). Anodic polarization curves of the electrode in 1.0 M NaCl + 0.1 M HF solution at the different surface ratio: (a) $S(\text{Fe}):S(\text{Ti}) = 1:1$; (b) $S(\text{Fe}):S(\text{Ti}) = 2:1$; (c) $S(\text{Fe}):S(\text{Ti}) = 5:1$. Key: 1 – Fe (electrode); 2 – Fe (component of the combined electrode); 3 – Ti (electrode); 4 – Ti (component of the combined electrode).
that HF concentration and the ratio of working surfaces of iron and titanium components affect the parameters of anodic polarization of the combined electrode.

In order to obtain a sufficient amount of the precipitate for further processing and analysis, electrolysis was carried out in fluoride concentrated solutions (in the HF concentration range from 0.1 to 1.0 mol/l) and at increased values of current density. The XRD patterns of the samples obtained by means electrolysis are shown in Figs. 6a and 6b. The results of X-ray diffraction study as a function of electrolyte composition, electrolysis parameters, surface ratio, heating temperature are summarized in Table 1 (the number of experiments in the table corresponds to the number of curve in Figs. 6a and 6b).

According to the data (Table 1), in the process of electrochemical synthesis in 1.0 M NaCl + 0.1 M HF solution (current density 100 mA/cm², S(Fe):S(Ti) = 2:1) the oxidation reaction with formation of a precipitate proceeds only on the iron component until its complete dissolution, while the titanium component does not dissolve.

The powdery product of brown-greenish color was formed after anodic polarization and subsequent filtration and drying for 8 h at 80°C. According to XRD analysis data (Fig. 6a, curve 1), for the dispersed material obtained using electrolysis and subsequent calcination, the positions of the diffraction lines only of iron oxide and oxyhydroxide are indicated. It should be noted, that according to data of quantitative phase content (Table 1), iron oxide and oxyhydroxide are formed with the structure in the percentage of magnetite (Fe₃O₄) 79.2 wt% and goethite (α-FeOOH) 20.8 wt%, respectively. After the high-temperature treatment (1100°C) only hematite α-Fe₂O₃ is presented in the powder composition (Fig. 6b, curve 1; Table 1).

In order to increase the dissolution rate of titanium component, the electrolysis conditions were changed – the fluoride content in the initial solution was brought to 0.5 mol/l and the surface area of iron relative to titanium was increased, S(Fe):S(Ti) = 5:1. Under these conditions, the dissolution of both components of the combined electrode was observed after 5 minutes of electrolysis, and one could see an intensive formation of a precipitate after 35 minutes. The dispersed material of saturated brown color was formed after its filtration and drying for 8 h at 80°C. The phase composition of this product is a mixture of iron hydroxides with goethite structure (93.6 wt%) and iron titanates of composition Fe(FeTi)O₄ (6.4 wt%) (Fig. 6a, curve 2; Table 1). After heat treatment (1100°C) hematite (92%) and iron titanates of composition Fe₃TiO₁₅ (8%) were formed in the precipitate (Fig. 6b, curve 2; Table 1).

In the following experiments, the polarization regime changed and the current density increased to 150-200 mA/cm² (experiments 3 and 4 in Table 1). Electrolysis at a current density of 150 mA/cm² promotes an increase in the intensity of dissolution of iron component (Table 1), however, the phase composition of the obtained sample remains practically unchanged (Fig. 6a, curve 3; Table 1). The high temperature treatment (1100°C) leads to the transition of iron titanates of composition Fe₃TiO₁₅ to their other form – Fe₂TiO₅ (7.6 wt%) (Fig. 6b, curve 3; Table 1).

A further increase in the current density up to 200 mA/cm² promotes a slight decrease in the intensity peaks corresponding to the phase of α-FeOOH (54.3 wt%) and an
increase in the content of the phase of Fe(FeTi)O$_4$ (13.3 wt%) (Fig. 6a, curve 4; Table 1). After high-temperature treatment (1100°C), one can see also a slight decrease in the content of the hematite phase (83.2 wt%) and an increase in the content of the phase of Fe$_2$TiO$_5$ (16.8 wt%).

Addition of HF (1.0 mol/l) to the initial solution of sodium chloride leads to a significant increase in the dissolution rate of titanium component (its value becomes comparable with the dissolution rate of iron component) and promotes a change in the phase composition of the obtained product (Table 1). XRD analysis shows (Fig. 6a, curve 5) the diffraction peaks of two phases – α-FeOOH (76.0 wt%) and TiO$_2$ (9.0 wt%). The increase in the calcination temperature to 1100°C leads to the ordering of the structure of the electrolysis product, and its phase composition is as follows: hematite (33.3 wt%) and Fe$_5$TiO$_4$ (62.5 wt%).

Figs. (7 and 8) compare the SEM images of titanium and iron surfaces after anodic polarization in the different electrolyte solutions. The surface of titanium component, formed in the process of electrolysis in sodium chloride solutions with the HF concentration ≥ 0.5 mol/l, becomes gray; the development of etch pits at the edges and on the surface of the metal is observed. Thus, titanium, in contact with iron, dissolves fairly evenly, but with the formation of a surface oxide film. Crystallites of the film have a clear facet up to 20 μm in size (Figs. 7a and 7b). The existence of this film is confirmed by the formation of a dense yellow coating on the surface. Under the same conditions, the dissolution of iron component is also accompanied by a more uniform change in the surface, which is covered with the products of dissolution without visible corrosion foci (Figs. 7c and 7d).

At a higher concentration of hydrofluoric acid (1 M), numerous local dissolution foci are present on the surface of
Fig. (7). SEM images of electrode surface of contacting metals after anodic polarization ($j = 100$ mA/cm$^2$, $t = 60$ min, $S$(Fe):$S$(Ti) = 5:1) in 1.0 M NaCl + 0.5 M HF solution: (a, b) Ti (in contact with Fe) and (c, d) Fe (in contact with Ti).

Fig. (8). SEM images of electrode surface of contacting metals after anodic polarization ($j = 100$ mA/cm$^2$, $t = 60$ min, $S$(Fe):$S$(Ti) = 5:1) in 1.0 M NaCl + 1.0 M HF solution: (a, b) Fe (in contact with Ti) and (c, d) Ti (in contact with Fe).
Fig. (9). (a-d) SEM image of the samples obtained by means of electrolysis (1.0 M NaCl+1.0 M HF, j = 100 mA/cm², temperature 25°C, duration of electrolysis 60 min, S(Fe):S(Ti) = 5:1) and subsequently calcined at 1100°C.
Fig. (10). (a-d) The data of SEM image of local analysis of the samples obtained by means electrolysis (1.0 M NaCl + 1.0 M HF, j = 100 mA/cm², temperature 25°C, duration of electrolysis 60 min, S(Fe):S(Ti) = 5:1) and subsequent calcined at 1100°C.

iron. They have a spherical shape with a diameter from 60-
100 μm to 150-200 μm (Figs. 8a and 8b). At the same time,
the surface of titanium is destroyed mainly along the edges
of the electrode, and destruction foci of different shapes are
formed, mainly rounded oblong (Fig. 8c) with a diameter of
10-20 μm, in the form of extensive ulcers (Fig. 8d).

Figs. (9a-d) show the particle morphology of the sample
obtained using electrolysis from the 1.0 M NaCl + 1.0 M HF
solution and subsequent thermal treatment. According to
these SEM images, the obtained precipitate has a very devel-
oped surface and submicron particle sizes. It is observed
(Figs. 9a-d) that particles of the sample are well-faceted
crystalline formations and consist of plates having nanoscale
facets and etching patterns on the lateral sides. The test sam-
ple consists of the units of different sizes and shapes,
preferably of irregularly shaped aggregates, with the pre-
dominance of hexagonal elongated and lamellar shape, and
also needle-shaped units. From the general view of precipita-
tion particles (Figs. 9a-d), aggregates of both relatively small
particles with size ~ 500 nm, and sufficiently large sizes -
about 7-14 μm can be seen.

The local analysis was performed in order to obtain addi-
tional and more detailed information about morphology,
structure, and chemical composition of the synthesized sam-
ple. These data (Figs. 10a-d) show the uneven distribution of
elements over the surface of the precipitate particles and indirectly confirm the previously given XRD analysis data.
According to the data of local analysis, the range of the iron
content (wt.%) is 20-70, while the content of titanium is an
average of 2-30 wt.%.

CONCLUSION

The joint electrochemical oxidation of electrically con-
tacting iron and titanium (as components of a combined elec-
trode) in aqueous solutions containing chloride and fluoride
ions was studied for the first time.

The possibility of synthesizing mixed dispersed oxide
system Fe₂O₃-TiO₂ using direct anode current in 1.0 M NaCl
+ x M HF solution (x = 0.5-1.0 M) and subsequent heat
treatment of the precipitate has been demonstrated. The
resulting product is a mixture of iron and titanium oxides,
but the work shows the possibility of controlling its
composition by changing the parameters of electrolysis, such
as current density, solution composition and concentration
ratio of the working surface area of metal components (iron
and titanium in the composition of the combined electrode).
At the same time, there is a clear correlation between these
electrolysis parameters and the composition and properties
of obtained precipitates and good reproducibility of the results.

A mixture of complex iron and titanium oxyhydroxides
(α-FeOOH – 93.4-93.6 wt% and Fe(FeTi)O₄ – 6.4-6.6 wt%)
has been formed during anodic dissolution of a combined
iron-titanium electrode in the 1.0 M NaCl + 0.5 M HF
solution (electrolysis conditions: j = 100-150 mA/cm², t = 60
min, S(Fe):S(Ti) = 5:1) and subsequent low-temperature
treatment (80°C). A mixture of α-Fe₂O₃ (33.3 wt%) and
Fe₂TiO₅ (62.5 wt%) has been formed under the same
conditions of electrolysis, but in solution with an increased
HF concentration (1.0 mol/l) and after the high temperature
treatment (1100°C).
The joint anodic dissolution of iron and titanium in halides solutions and subsequent hydrolysis reaction makes it possible to obtain crystalline structures of complex titanium and iron oxyhydroxides at relatively low temperatures. The synthesized products can be used as precursors in the process of producing ceramics and composite materials.

CONSENT FOR PUBLICATION
Not applicable.

AVAILABILITY OF DATA AND MATERIALS
Not applicable.

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CONFLICT OF INTEREST
The authors declare no conflict of interest, financial or otherwise.

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