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THE ELECTROCHEMICAL FACING OF POWDER IRON FOR THERMOGALVANIC ELEMENTS

Background. The oxygen impact for electrochemical formation of surface structures on iron particles is not sufficiently taken into account in the production technology of composite electrodes for thermogalvanic energy sources; the main conditions of the performance improvement are considered.

Objective. The aim of the paper is to define the electrochemical conditions of oxide layers formation on the surface of the iron powder composite electrode with the presence of oxygen in neutral and basic media.

Methods. For better performance of the oxygen impact the study of composite electrodes was conducted in open air and inert atmosphere, the assembly of cells with composite electrodes was carried out on air in two versions: hermetic and perforated. Electrochemical studies were carried out on the electrochemical module Autolab 30 PGSTAT301N Metrohm Autolab using 3-electrode cells. The microtexture of the iron powder during redox reactions was examined using a high resolution scanning electron microscope Mira 3 FESEM Tescan USA Inc. on a cathode with field emission at SEM HV-10 KeV and automatic measurement on the image. The analytical regime of the scanning microscope was used to determine the surface components distribution and their degrees of oxidation.

Results. It was determined that with a low oxygen partial pressure in potential range from -0.23 to -0.88 V surface iron structure has two stages of organization: 1 – mosaic stage with partial covering of iron surface FeO and Fe₂O₃; 2 – transformation of mosaic structure into core–shell structure. Under a high oxygen partial pressure and cathodic potential >-0.88 V thick iron oxide layers have been formed on the surface structure of iron particles. Thus, the oxygen partial pressure has a key role in electrochemical formation of nanolayers on the surface of iron particles and realizes the thermoelectrochemical activity of composite electrodes.

Conclusions. The study found that thermoelectric parameters of an electrode based on iron powder are determined as a function of the pH of the medium and the oxygen content. In the neutral medium electrodes lose ability to reversible processes needed for the formation of the thermogalvanic properties. The ability of a powdered iron-composite electrode to a thermo-galvanic element is formed in an alkaline medium.

Keywords: iron; carbon; composite electrode; oxygen; coatings; electrochemical formation.

Introduction

Now, significant attention has been assigned to the design and controlled synthesis of semiconductive nanomaterials representing particular functional characteristics such as thermoelectric activity in narrow temperature range. In this case a correlation of *n*- and *p*-types of oxides is the main factor of the thermal gradients formation. Hence the regulation of the oxide types and thickness of their surface oxide layers become one of the main goals connected with the production of a new generation of thermoelectric and thermogalvanic cells. If the defects act as the dopants, oxygen vacancies and cation vacancies have been indicated *n*-type and *p*-type conductivity, respectively. So, Fe-ferric oxide systems can be used for such purposes due to their ability for changing of the conductivity type in the presence of oxygen and reductant. The first key

parameter in describing the surface organization of iron particles is transport properties of oxygen and cation vacancies. Hence the kinetics of surface film growth is the vacancy diffusivity (D_o), which appears in the Nernst–Planck equation for the flux and temporal dependence of the vacancy concentration. Typically, the oxide layer of iron surface consists of the magnetite (Fe₃O₄) or a mixture of magnetite and maghemite (γ -Fe₂O₃) [1]. Thick iron oxide layers may contain multiple metal and oxide layers, i.e. Fe; FeO; Fe₃O₄; Fe₂O₃ [2]. More over Fe₂O₃ and γ -Fe₂O₃ have a similar spinel crystal structure with only a small difference in the lattice constants. Such local surface combination of two conductivity types should lead to the appearance of new electrochemical and thermochemical properties of electrode/electrolyte interface. Thus the presence of oxidizes (particularly O₂) and reductants (e.g., carbon) of iron and iron oxides can ap-

pear for regulator of surface structure and change types of conductivity. In all these cases the volume chemical reactions have been used for formation of surface oxides. Such technologies have one main limitation – the absence of the surface thickness layer control. On another hand, the electric current also can be use as oxidizing or reducing agent. In this case the thickness and composition of the surface oxide film depends on applied potentials, pH of the electrolyte and kinetic of the current passed through the cell [3, 4]. All these parameters are well controlled and give an opportunity to receive the surface layers with given properties [4, 5].

Problem statement

The aim of the paper is to study electrochemical conditions of oxide layers formation on the surface of the iron powder electrode with the presence of oxygen in neutral and basic media.

Experimental

A predetermined amount of the iron powder (PGR 3.200.28-30) with bulk density $2.7 \pm 0.2 \text{ g/cm}^3$ (Ukraine), activated carbon BAU-A (Ukraine) (Table 1) and 1% solution of polyvinylidene fluoride (PVDF) in acetone (Sigma-Aldrich) were stirred by ultrasonic mixer to homogeneity. Then, electrode mass was pressed on steel grid with size of 1 cm^2 .

Electrolyte solution was prepared from sodium hydroxide (NaOH) pellets (Sigma-Aldrich, minimum 99.0% purity), sodium chloride (NaCl) (Sigma-Aldrich $\geq 99\%$, AR grade) and distilled water.

Table 1. Content of the iron powder and carbon in the active electrode mass composition

$m(\text{Fe}), \text{ g}$	$m(\text{C}), \text{ g}$	$\Sigma m, \text{ g}$
0.106	0.046	0.152
0.149	0.0560	0.205
0.159	0.0542	0.213

The assembly of cells with composite electrodes was carried out on air in two versions: hermetic and perforated. The electrode mass and separator were saturated with the 5 M solution of NaOH in water. The thickness of anode provided the dimensions necessary for disk elements in the standard 2016. After that, the elements were hermetically sealed. For the probability of contact with air, some covers were perforated before sealing.

Electrochemical studies were carried out on Autolab 30 PGSTAT301N Metrohm Autolab electrochemical module using 3-electrode cells. The powdered iron was used as working electrode, as counter electrodes were platinum (Pt) and carbon (C) and Ag/AgCl – reference electrode. Cyclic voltammograms (CVA) and open circuit potential (OCP) data were recorded at the potential scan rate of 0.1 and 50 mV/s in 0.1, 5 M solutions of NaOH and 1 M NaCl. Resistance of cells was determined on Autolab 30 PGSTAT301N Metrohm Autolab electrochemical module, in the two-electrode cell (disk cell) in the frequency range 10^{-2} – 10^6 Hz. Current (I) was calculated using Ohm's law. Power-to-weight output was calculated by $P = IV/m$, where I – current, V – OCP, m – weight of the active electrode mass.

The microtexture of the iron powder during redox reactions was examined using Mira 3 FESEM Tescan USA Inc. high resolution scanning electron microscope on a cathode with field emission at SEM HV-10 KeV and automatic measurement on the image. The analytical regime of the scanning microscope was used to determine the surface components distribution and degrees of oxidation.

Thermal impact was tested using electromechanical middle temperature thermostat with lead accuracy $\pm 1 \text{ }^\circ\text{C}$.

Results and Discussion

CVA of the iron powder electrode in neutral and basic media have considerable differences in cathodic and anodic wave shapes. Also they have the different potential ranges of stability (Fig. 1, *a*, *b*). In the neutral media such range is 0,5–0,75 V with surface passivation in wide potential range -0.76 – $+1,9$ V via NHE (Fig. 1, *a*). In the basic media the potential range of stability increased up to 1.2–1.4 V without any passivation (Figs. 1, *b* and 2, *a*).

By logarithmic dependences of current changes differences in electrochemical behavior of electrodes were specified (Fig. 2). According to Tafel equations composite electrodes have only one zero net current potential at -0.173 V with further dissolution in wide potential range from -0.5 to -2.29 V in neutral media (Fig. 2, *a*), but in basic media such system has two zero net current potentials at -0.68 and $+0.35$ V with passivation range between -0.23 and -0.88 V (Fig. 2, *b*) [5, 6].

Using the theory of mixed potentials based on the independence of the partial anodic and cathodic reactions, it can be assumed that more than

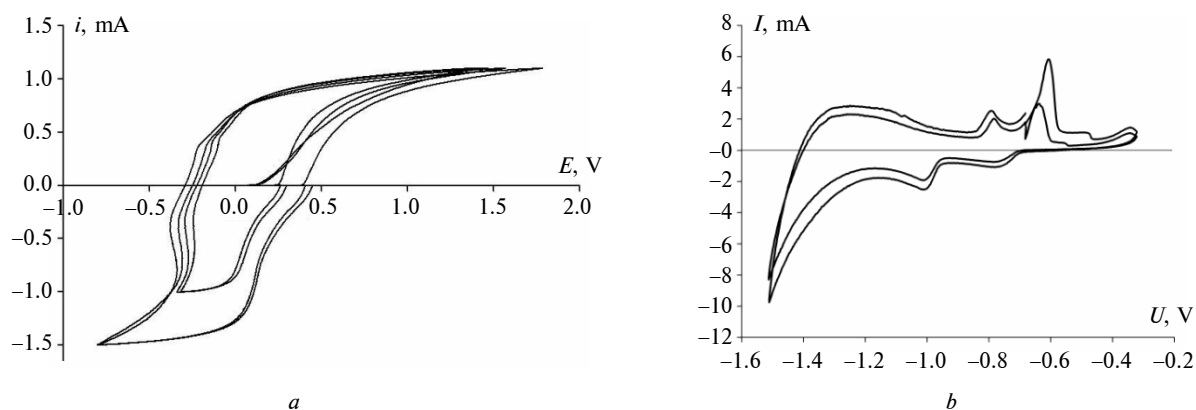


Fig. 1. CVA curves of iron powder electrode: *a* – in 1 M NaCl (neutral media), *b* – in 0,1 M NaOH (basic media)

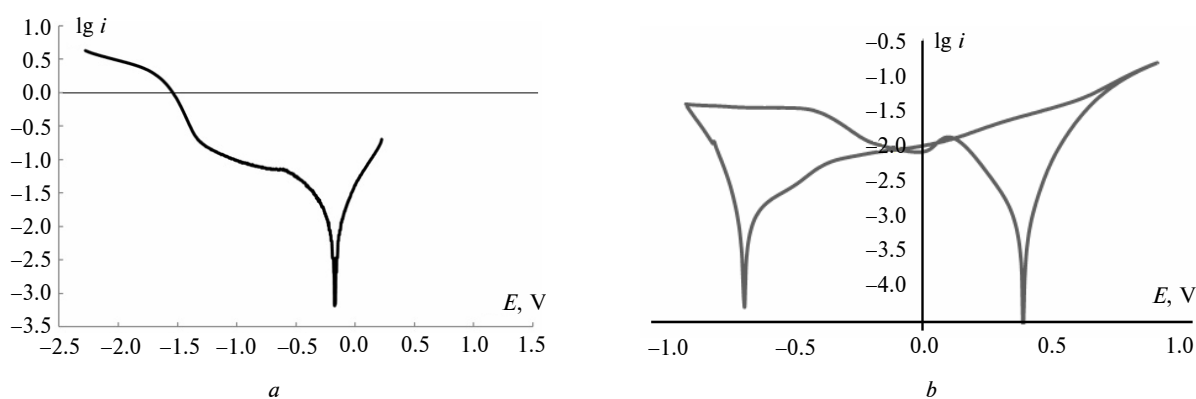


Fig. 2. Polarization curves of iron powder electrode in: *a* – 1 M NaCl; *b* – 0,1 M NaOH

Table 2. Main standard potentials of iron redox reactions in water media [10]

№	Electrode reaction	Standard potential via NHE, V
1	$\text{Fe}_3\text{O}_4 + \text{H}^+ + e \rightarrow \text{FeO} + 8\text{H}^+$	-0.173
2	$\text{HO}_2^- + \text{H}_2\text{O} + e \rightarrow \text{OH}(\text{aq}) + 2\text{OH}^-$	-0.245
3	$\text{Fe}(\text{OH})_3 + e \rightarrow \text{Fe}(\text{OH})_2 + \text{OH}^-$	-0.56
4	$\text{Fe}(\text{OH})_2 + \text{OH}^- \rightarrow \text{FeOOH} + \text{H}_2\text{O} + e$	-0.56
5	$\text{O}_2(\text{g}) + e \rightarrow \text{O}_2^-$	-0.563
6	$\text{FeO}_2^- + e \rightarrow \text{FeO}_2^{2-}$	-0.68
7	$[\text{Fe}_2(\text{OH})_6(\text{OH}_2)_3]_n^{2-} + 3n\text{OH}^- \rightarrow [\text{Fe}_2\text{O}_3(\text{OH})_3(\text{OH}_2)_3]_n^{3-} + 3n\text{H}_2\text{O} + 2ne^-$	-0.88
8	$3\text{Fe}^{3+} + 4\text{H}_2\text{O} + e \rightarrow \text{Fe}_3\text{O}_4 + 8\text{H}^+$	+0.35

one electrochemical reaction takes place simultaneously on the composite electrode surface [7–9]. According to value of potential on the ferric surface the reaction 1 (Table 2) can take place in the neutral media. More over in all regions (cathodic and anodic) electrode has tendency to dissolve iron from their surface. So, ferric composite electrode can't have enough driving force for reversible redox reactions of the chemical energy transformation [7].

On the other hand, if the pH medium is not basic enough, ferric compound may be dehydrated with generation of a ferric oxide without high concentration of oxygen and cation vacancies. In such case the ability of thermoelectric conversion of material disappears [3, 4]. In the basic media the ability of the composite electrode charging increases due to presence of two zero net current potentials corresponding to reactions 6 and 8.

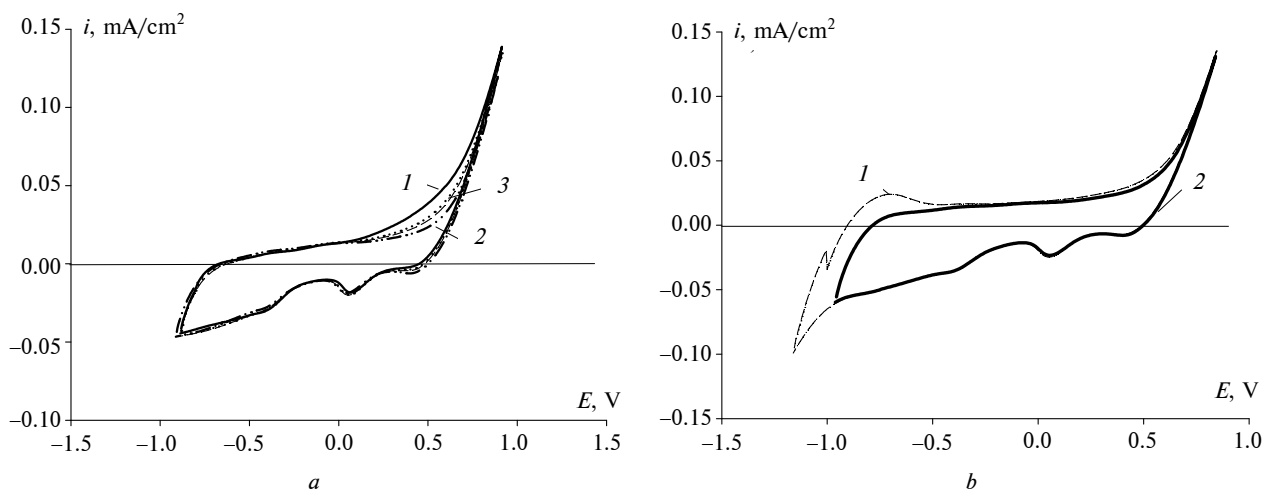
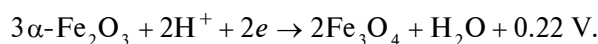


Fig. 3. CVA curves of iron powder electrode polarized via: *a* – Pt electrode: 1 – first cycle, 2 – second cycle, 3 – cycles 3–10; *b* – carbon electrode: 1 – first cycle, 2 – cycles 2–10

Also occurrence of three peaks on CVA curve at potentials -0.245 , -0.56 , and -0.88 V initiated formation of the various ferric hydroxides. However, if the pH is around 8 or 9, ferric hydroxide can be reduced at the cathode to form Fe_3O_4 as represented in the equation 8 (see Table 2). According to [7], the formation of a polymeric microdispersed hydrous oxide layer on the iron surface in alkaline media (pH 10–12) can be expected during cycling of the iron electrode (eq. 7). Reaction (7) initiates hydrous oxide films formation on the oxide/solution interface (see Fig. 1, *b*). This interface has compound structure and consists of an inner compact anhydrous layer MO_x and an outer microdispersed hydrous layer of general form $\text{MO}_a(\text{OH})_b(\text{OH}_2)_c$.

According to Tafel equations such structure starts to generate at -0.23 V via NHE. Maximum film thickness is obtained at -0.9 – 1.0 V. So on powdered iron/solution interface hydrous oxide layers are generated during cycling in the potential range from -0.23 to -1.0 V via NHE.

CVA curves of composite electrodes polarized relatively platinum and carbon have no differences in peak potentials in basic media (Fig. 3). But the first cycle of the composite electrodes under polarization relatively carbon found non stable behavior (potential range -1 – $+1$ V) in comparison with further cycles (Fig. 3, *b*). So, the first cycle is the cycle of the formation of new surface structure in the Fe-C-NaOH electrochemical system. More over the new peak appears at potential $+0.22$ V. According to standard potentials of iron redox reactions in water media of such potential is the potential of the reaction [10]:



Hereby, the formation of iron oxides mixture (Fe_3O_4) in basic media on the particle surface takes place during cycling of composite electrode in potential range -1 – $+1$ V via NHE.

Taking into account various structures of iron oxides (the FeO has layer structure and all modifications of Fe_2O_3 have spinel crystal structure) the structure of electrode surface would be complicated under the cycling [11–14].

SEM analysis of the electrode samples after 1 cycle found decreasing of amount of pure iron (Fe^0) on 0.24 mas. % and covering of the particles surface with spherical and layer structures (Fig. 4, *a*) like mosaic. Cycling under limited presence of oxygen brings the surface into a state of increasing of particle size with retention of the mosaic surface structure (Fig. 4, *b*). If the electrode surface has the long contact with air, such mosaic structure became to monolayer structure (Fig. 4, *c*). In this case the formation of the core–shell structure takes place.

The common scheme of the electrode surface transformation in basic media at potential range from -0.23 to -1 V has two stages: 1 – mosaic stage with partial covering of iron surface FeO and Fe_2O_3 ; 2 – transformation of mosaic structure into shell-core structure (where a shell_A is the FeO, core is an iron particle) or shell_A-shell_B-core structure (where shell_B is the mixture of FeO and Fe_2O_3).

Such oxide mixture on the iron surface is conditional on the formation of contacting *n*-type and *p*-type semiconductors with various ther-

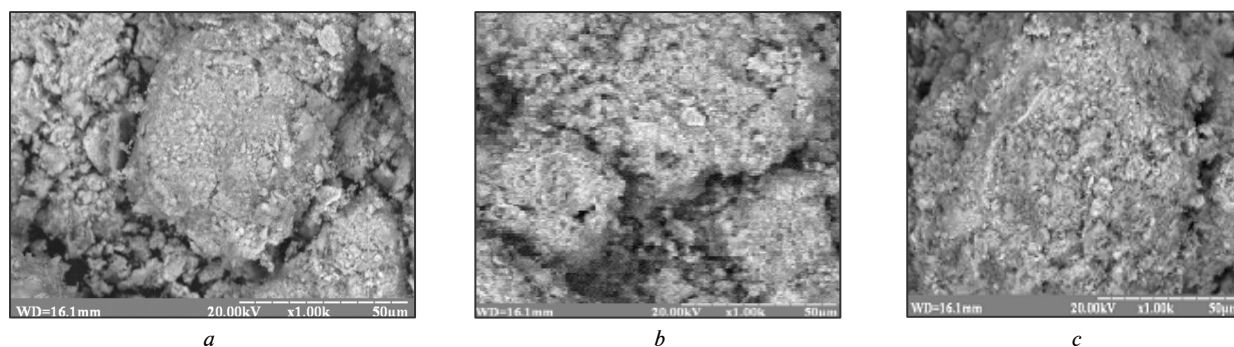


Fig. 4. Microphotos of the electrodes surface in the potential range from -0.23 to -1 V: *a* – 1 cycle; *b* – under the limited presence of oxygen; *c* – at the long contact with air

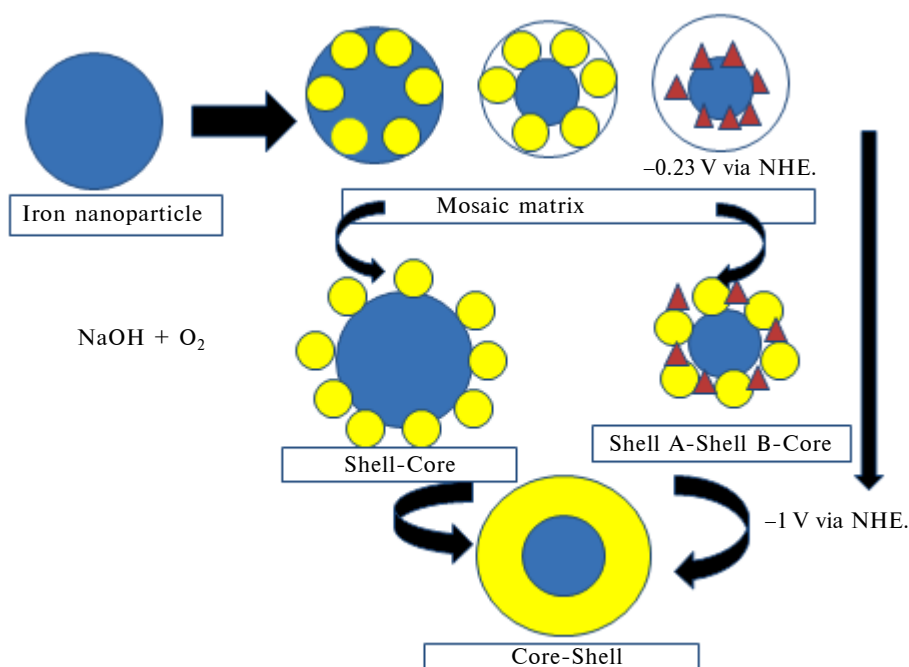


Fig. 5. Scheme of iron particles surface organization in various conditions

Table 3. Thermoelectric coefficients of hermetic and perforated thermogalvanic elements

Mass of Fe, g	OCP, V	i_0 , A	Power-to-weight, W/g	Conditions
0.1481	3.51 ± 0.01	0.081 ± 0.001	1.89 ± 0.04	Hermetic
0.1518	4.02 ± 0.01	0.25 ± 0.001	6.58 ± 0.04	Hermetic
0.1481	0.08 ± 0.01	0.041 ± 0.001	0.022 ± 0.005	Perforated
0.1518	0.03 ± 0.01	0.083 ± 0.001	0.016 ± 0.005	Perforated

moelectric properties. So it caused the occurrence of the thermo electromotive force and thermal gradients in the contact place. If the surface of electrode has a long contact with air such structures transform into core–shell structure (Fig. 5). In this case the oxygen partial pressure increased and thick iron oxide layers formed on iron surface with low ability to vacancy diffusion [4, 15].

Further testing of the disk elements with composite electrodes proved the oxygen dependence of the thermoelectric power density (power-to-weight). In hermetic elements such density was higher than in contacted air (perforated) elements. So, the thick oxide layer on the surface of iron particles blocked thermoelectric activity of the thermogalvanic elements. After the long contact

with air perforated elements have lost their thermoelectric activity while hermetic elements have retained this activity for 8 months (Table 3).

Conclusions

The comprehensive study has shown key factors of formation of the composite powdered iron electrode thermochemical activity. The thermoelectric parameters are defined as a function of pH medium and oxygen content. In the neutral media electrodes lose ability to reversible processes needed for the formation of the thermogalvanic properties. Such ability is formed in alkaline media. Under high oxygen partial pressure and cathodic potential > -0.88 V thick iron oxide layers have been formed on the surface structure of iron particles. In low oxygen partial pressure in potential range from

-0.23 to -0.88 V surface iron structure has two stages of organization: 1 – mosaic stage with partial covering of iron surface FeO and Fe₂O₃; 2 – transformation of mosaic structure into core–shell structure (where a shell structure consists of the FeO and core is an iron particle) or shell_A–shell_B–core structure (where shell is the mixture of FeO and Fe₂O₃). So, the oxygen partial pressure has a key role in electrochemical formation of nanolayers on the surface of iron particles and realizes the thermoelectrochemical activity of composite electrodes.

The results will extend the range of application of thermo-galvanic elements with composite electrodes by increasing their efficiency and reducing cost. Also, these electrodes are promising for use in iron-oxygen batteries.

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ЕЛЕКТРОХІМІЧНЕ ПОКРИТТЯ ПОРОШКОВОГО ЗАЛІЗА В ТЕРМОГАЛЬВАНІЧНИХ ЕЛЕМЕНТАХ

Проблематика. Вплив кисню на електрохімічне формування поверхневих структур на частинках заліза недостатньо враховується в технології виробництва композитних електродів для термогальванічних джерел струму; розглядаються основні умови підвищення продуктивності.

Мета дослідження. Мета роботи полягає в тому, щоб визначити електрохімічні умови формування оксидних шарів на верхній композиційного електрода із залізного порошку за наявності кисню в нейтральних і основних середовищах.

Методика реалізації. Для підвищення ефективності впливу кисню дослідження композитних електродів проводилося на відкритому повітрі та в інертній атмосфері, монтаж комірок із композитними електродами здійснювався на повітрі у двох варіантах: герметичному і перфорованому. Електрохімічні дослідження проводилися на електрохімічному модулі Autolab 30 PGSTAT301N Metrohm Autolab із використанням 3-електродних комірок. Мікротекстуру залізного порошку під час окисно-відновних реакцій досліджували з використанням скануючого електронного мікроскопа з високою роздільною здатністю Mira 3 FESEM Tescan USA Inc. на катоді з польовою емісією при SEM HV-10 кеВ і автоматичним вимірюванням на зображенні. Аналітичний режим скануючого мікроскопа використовувався для визначення розподілу компонентів поверхні та ступеня їх окиснення.

Результати дослідження. Встановлено, що за низького парціального тиску кисню в діапазоні потенціалів від $-0,23$ до $-0,88$ В поверхнева структура заліза має дві стадії організації: 1 – мозаїчна стадія із частковим покриттям поверхні заліза FeO і Fe₂O₃; 2 – перетворення мозаїчної структури на структуру ядро–оболонка. За високого парціального тиску кисню і катодного потенціалу $> -0,88$ В на поверхні структури частинок заліза утворилися товсті шари оксиду заліза. Таким чином, парціальний тиск кисню грає основну роль в електрохімічному формуванні наночарів на поверхні частинок заліза і реалізує термоелектрохімічну активність композитних електродів.

Висновки. У ході дослідження було встановлено, що термоелектричні параметри електрода на основі порошкового заліза є функціями рН середовища і вмісту кисню. У нейтральному середовищі електроди втрачають здатність до оборотних процесів, необхідних для формування термогальванічних властивостей. Здатність порошкового залізо-композитного електрода до термогальванічного елемента формується в лужному середовищі.

Ключові слова: залізо; вуглець; композитний електрод; кисень; покриття; електрохімічне формування.

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ЕЛЕКТРОХИМИЧЕСКОЕ ПОКРЫТИЕ ПОРОШКОВОГО ЖЕЛЕЗА В ТЕРМОГАЛЬВАНИЧЕСКИХ ЭЛЕМЕНТАХ

Проблематика. Воздействие кислорода на электрохимическое формирование поверхностных структур на частицах железа недостаточно учитывается в технологии производства композитных электродов для термогальванических источников тока; рассматриваются основные условия повышения производительности.

Цель исследования. Цель работы состоит в том, чтобы определить электрохимические условия образования оксидных слоев на поверхности композиционного электрода из железного порошка при наличии кислорода в нейтральных и основных средах.

Методика реализации. Для повышения эффективности воздействия кислорода исследование композитных электродов проводилось на открытом воздухе и в инертной атмосфере, сборка ячеек с композитными электродами проводилась на воздухе в двух вариантах: герметичном и перфорированном. Электрохимические исследования проводились на электрохимическом модуле Autolab 30 PGSTAT301N Metrohm Autolab с использованием 3-електродных ячеек. Микротекстуру железного порошка во время окислительно-восстановительных реакций исследовали с использованием сканирующего электронного микроскопа высокого разрешения Mira 3 FESEM Tescan USA Inc. на катоде с полевой эмиссией при SEM HV-10 КэВ и автоматическим измерением на изображении. Аналитический режим сканирующего микроскопа использовался для определения распределения компонентов поверхности и степени их окисления.

Результаты исследования. Определено, что при низком парциальном давлении кислорода в диапазоне потенциалов от $-0,23$ до $-0,88$ В поверхностная структура железа имеет две стадии организации: 1 – мозаичная стадия с частичным покрытием поверхности железа FeO и Fe₂O₃; 2 – превращение мозаичной структуры в структуру ядро–оболочка. При высоком парциальном давлении кислорода и катодном потенциале $> -0,88$ В на поверхности структуры частиц железа образовались толстые слои оксида железа. Таким образом, парциальное давление кислорода играет основную роль в электрохимическом образовании нанослоев на поверхности частиц железа и реализует термоэлектрохимическую активность композитных электродов.

Выводы. В ходе исследования было установлено, что термоэлектрические параметры электрода на основе порошкового железа определяются как функция рН среды и содержания кислорода. В нейтральной среде электроды теряют способность к обратимым процессам, необходимым для формирования термогальванических свойств. Способность порошкового железно-композитного электрода к термогальваническому элементу формируется в щелочной среде.

Ключевые слова: железо; углерод; композитный электрод; кислород; покрытия; электрохимическое образование.

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