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RESEARCH ARTICLE

Manufacturing of nanopillar (ultra-dispersed) catalytically active materials through chemical engineering

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Abstract

Objectives. Catalytically active materials are required in different chemical engineering processes. This makes the development of new materials with high efficiency and original ways in which to obtain them of significant interest. The present work investigates the synthesis of catalytically active material including electrode materials, as well as their improved efficiency due to the nanodecoration of their surface.

Methods. An aluminum folio was nanoperforated (nanoscalloped) by high-voltage anodization in an acidic medium. The effective electrode material was obtained as a metallic nickel replica rather than an oxide layer of the product. To study the surface state of aluminum obtained in this manner, a scanning electron microscope (Hitachi-SU8200) was used. The elementary composition of the aluminum was determined by back-scattered X-ray irradiation.

Results. The nickel replica obtained in the above-described process exceeded the catalytic activity estimated by methanol oxidation of the unprocessed nickel 70–150 times.

Conclusions. The present paper demonstrates the potential of creating effective catalytically active nanopillar materials using the metallic rather than metal-oxide part of a layer of anodized aluminum as a matrix template.

Keywords: distillation, binary mixtures, relative volatility, reflux ratio, distribution coefficient, internal energy saving in distillation

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НАУЧНАЯ СТАТЬЯ

Химико-технологический подход к созданию нановорсистых (ультрадисперсных) каталитически активных материалов**А.П. Антропов, Н.К. Зайцев, Е.Д. Рябков, Н.А. Яштулов[@], П.Н. Мудракова***МИРЭА – Российский технологический университет (Институт тонких химических технологий им. М.В. Ломоносова), Москва, 119571 Россия**[@]Автор для переписки, e-mail: yashtulovna@mail.ru***Аннотация**

Цели. Каталитически активные материалы остаются востребованными в различных химико-технологических процессах, поэтому актуальными являются исследования, направленные на поиск новых эффективных материалов и оригинальных путей их получения. Настоящая работа посвящена созданию ленточных каталитических, в том числе электродных материалов, эффективность которых увеличена за счет нанорифления поверхности.

Методы. Методом высоковольтной анодной обработки на поверхности алюминиевой фольги формировалось нанорифление. Эффективный каталитически активный материал получали как никелевую реплику с металлической алюминиевой ленты. Для определения состояния поверхности алюминия использовали сканирующий электронный микроскоп Hitachi-SU8200 (Япония), для элементного анализа состава поверхности – обратную рентгеновскую фотоэлектронную микроскопию.

Результаты. Полученный нановорсистый никелевый материал превосходит по каталитической активности гладкий никель при окислении метанола в 70–150 раз.

Выводы. Продемонстрирована возможность использования в качестве темплатной матрицы для создания эффективных нановорсистых никелевых ленточных катализаторов, в том числе электродов, не алюминий-оксидной (как предлагалось ранее), а металлической части алюминиевой фольги, подвергнутой высоковольтному анодированию.

Ключевые слова: высоковольтное анодирование алюминия, нанорифление поверхности, ленточные катализаторы, нановорсистые электроды, ультрадисперсные электроды

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INTRODUCTION

Modern chemical technology enables the production of not only substances of a specific composition and structure, but also materials with a given dispersion and surface conditions that can serve as effective catalysts, afterburning catalysts for automobile exhaust, high-performance electrode materials for electrolyzers, and as chemical current sources. Chemical–technological approaches show great promise for creating materials with specific surface profiles because mechanical or thermophysical methods are not applicable in this case. Such materials include metal strips and

plates with guaranteed roughness in the form of micro- and nanoscale needles or columns, so-called nanofilaments, or ultrafine materials, which are characterized by high catalytic activity. These materials can be used in numerous applications in small-tonnage production [1–13] but are typically not employed in large-tonnage production. The reason for this is because nanofilament materials are obtained in the form of samples with an area of several square centimeters only, making samples of a sufficiently large area unavailable.

Increasing demands regarding environmental, information, and technological security aspects have necessitated the search for new catalysts and

alternative energy sources, based on using renewable resources and enabling the more efficient use of non-renewable resources, as well as finding autonomous power sources for various technical devices and aircraft. The task of increasing the efficiency of fuel cells and water electrolysis systems is relevant for two primary reasons. First, the systems of electrolyzer fuel cells represent the most real energy buffers required for the operation of alternative energy sources, common among which is variable power. Effective operation of such energy sources is impossible without energy buffers; that is, systems that allow for shading (smoothing) or compensating for fluctuations in the power of the original energy sources.

The first stage in the manufacturing of nanofilament materials is the single/double relatively high-voltage anodic treatment of aluminum or titanium surfaces with suitable electrolytes, among which the most commonly used are oxalic, sulfuric, and phosphoric acids. In this case, an oxide layer is formed on the surface of aluminum, comprising hexagonal cells arranged in a honeycomb formation and permeated with nanopores throughout. Paszanski and Schneider [14] suggested the emergence of a dissipative microcirculation system similar to Rayleigh–Benard cells, where on the surface of aluminum the electric potential gradient acted instead of the temperature gradient, and the diffusion process instead of Archimedean force. In the case of aluminum anodizing, the oxide layer comprises aluminum hydroxide nanoparticles in various degrees of hydration that have pores with a very large aspect ratio; this pore diameter can be up to 1 nm, and its length can be up to several microns. Concurrently, the surface of metallic aluminum is also grooved, which reflects uneven propagation of the oxidation front deep into the aluminum plate [15]. The fluting of aluminum is much smaller in aspect ratio compared with that in the oxide layer; however, unlike the aluminum-oxide membrane, which does not have mechanical strength, metal tape can be used in a tape-drawing mechanism, as well as storage and transportation in a rolled state, and is much more technologically advanced for creating large-area nanofilament matrices. In all of the publications we found [1–13], the use of an aluminum-oxide layer as the initial (template) matrix was described; the typical samples that the authors of these works worked with were several square centimeters in size. The question on the possibility of the practical use as a starting matrix for the creation of functional electrodes the metal part of anodized aluminum instead of aluminum-oxide remains unexplored.

This work investigated the possibility of obtaining nanofilament nickel replicas with a higher catalytic activity compared with smooth nickel,

using a replica method not based on an aluminum-oxide layer but using metallic aluminum. Finding a solution to this problem can help to determine the feasibility of developing chemical and technological installations for creating initial (template) matrices based on aluminum tape.

EXPERIMENTAL

The formation of aluminum matrices was performed on aluminum foil samples with a purity of 98.5%; 0.3 M orthophosphoric acid (analytical reagent (AR) grade) was used as a background electrolyte. Oxygen was removed from the solution by purging spectrally pure argon (American Chemical Society (ACS) grade) and during anodization, gas purging continued. The auxiliary electrode was a platinum wire with a diameter of 0.5 mm and a length of 10 cm. A potentiostat with an operating voltage range from 0 to 300 V was used as a voltage source; a maximum current of up to 10 A was allowed and the accuracy of maintaining the voltage at 0.5 V was ensured. A sample of aluminum foil with an uninsulated area of 2 cm² was placed in a cell (a 500 mL laboratory beaker) equipped with a RITM-01 magnetic stirrer (*Econix-Expert*, Moscow, Russia). The aluminum-oxide layer for obtaining a purified aluminum surface was removed by placing in a 0.1 M sodium carbonate solution (AR) for 30 min at 100°C.

The sample processed during the selected time and at the selected voltage was washed with bi-distilled water and dried in air for 24 h. An enlarged image of the surface of aluminum was obtained using an optical microscope, photographed, and examined on a Hitachi-SU8200 scanning electronic microscope (SEM; *Hitachi*, Japan) in a low-voltage low-temperature mode (at accelerating voltages of 15 kV and 77 K). Scanning electron microscopy was the main method for determining the number and size of holes (depressions) on the surface of the aluminum. In addition, reverse X-ray photoelectron microscopy was used for elemental analysis of the surface composition. The primary image processing of electronic micrographs was performed using the Digimizer (v.5.4.7) software program.

As catalytically active surface modifiers, palladium nanoclusters were synthesized from palladium dichloride (AR) through water microemulsions in octane (AR) stabilized with a non-ionic surfactant (Triton X-100), followed by the reduction of palladium ions with sodium tetrahydroborate (AR), similar to the process effected in our previous works [16–18]. For the preparation of nickel replicas, nickel plating solutions containing 150 g/L of nickel nitrate (AR), 80 g/L ammonium sulfate (AR), and 50 g/L of the disodium salt of ethylenediaminetetraacetic acid

(AR) was used. Nickel plating was carried out in the same electrochemical cell as anodization but a cathode voltage of -5 V was applied to the sample. Copper replicas were similarly prepared. The composition of the copper plating electrolyte was as follows: 80 g/L copper sulfate pentahydrate (AR), 80 g/L sodium thiosulfate (AR), and 80 g/L sodium acetate (AR). The time taken for the electrochemical reduction of metals to the surface of the nanoporous membrane for each of the samples was 120 min.

After applying the replicas to the surface of the nanoporous matrix, the aluminum template material was removed by keeping the resulting “sandwich” for 24 h in a 4 M solution of sodium hydroxide (AR), which was then repeatedly washed with bi-distilled water, and dried in air for 24 h at 21°C . Following on, the replicas were investigated by optical and electron microscopy as described above.

To study the electrochemical activity of the obtained replicas, we used the cyclic voltammetry method on the Ecotest-VA (*Econix-Expert*) voltammetric analyzer. In the case of nickel replicas, aqueous methanol solutions were used as the main substrate. For copper replicas, aqueous glucose solutions were used. The specific composition of the background electrolytes is indicated on the signatures of the voltammograms.

RESULTS AND DISCUSSION

The formation of the anode layer at the initial stage of anodization (15 min after the start of the process) is shown in Fig. 1. As can be seen from the figure, light contours—the boundaries of the initial metal crystallites—appeared on the sample surface. The black dots of micropore nuclei were evenly distributed over the sample surface, which is consistent with Thompson’s observations [9].

With further anodization, dimples formed on the metal surface. The dependence of the average pore size on the surface of the aluminum foil on the applied voltage and the anodizing time is shown in Fig. 2.

As shown, the average size of the pores that represent the imprint of the propagation front of the Keller layer depends not only on the magnitude of the applied voltage but also on the exposition time. This suggested that the dynamic evolution of the anodized layer occurred not only at the boundary of the Keller and Thompson layers (as Thompson suggested [19]) but also at the Keller metal-layer boundary. During further anodization, depressions formed on the metal surface [20, 21].

The dependence of the current density on time at a fixed voltage (70 V) during the sample’s anodization process is shown in Fig. 3.

Figures 4 and 5 show SEM images of aluminum foil samples subjected to anodization at 70 V in 0.3 M phosphoric acid after removing the aluminum-oxide

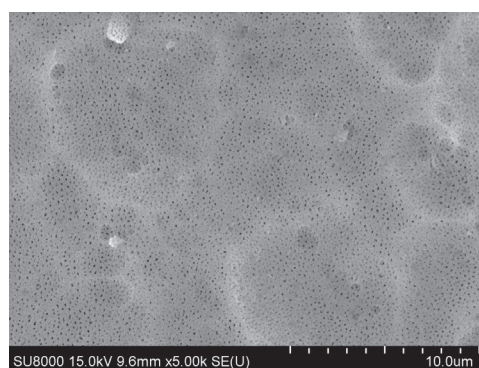


Fig. 1. Scanning electronic microscope (SEM) micrograph of an aluminum sample after anodization ($U = 90$ V, $t = 15$ min, $T_{\text{el}} = 21^{\circ}\text{C}$, 0.3 M H_3PO_4).

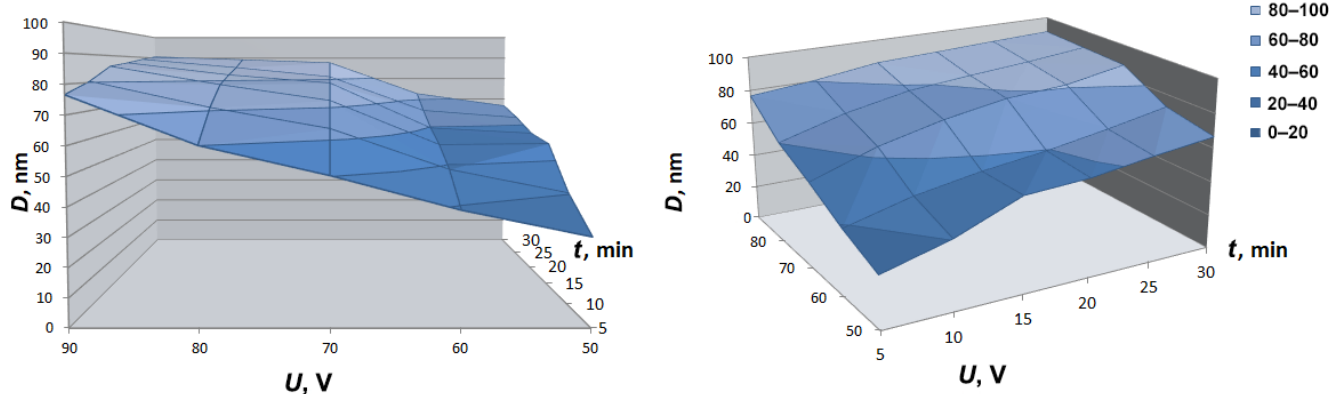


Fig. 2. A three-dimensional diagram of the dependence of the average diameter of holes formed on the aluminum surface during anodization on the voltage and time of anodizing ($T_{\text{el}} = 21^{\circ}\text{C}$, 0.3 M H_3PO_4).

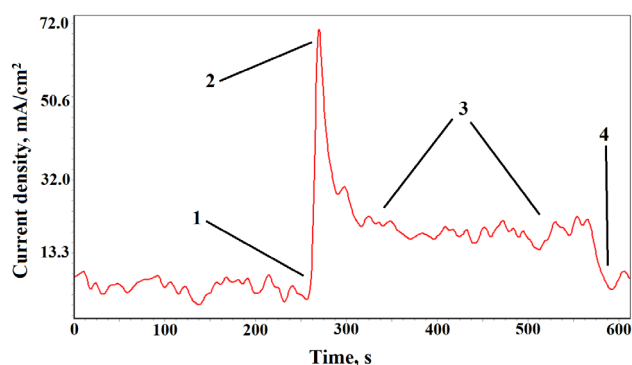


Fig. 3. Change in the current density during anodization in the mode of 70 V, 5 min, $T_{el} = 21^{\circ}\text{C}$ in 0.3 M H_3PO_4 : (1) the start of the anodization process (voltage supply); (2) the formation of an aluminum-oxide layer; (3) electrical breakdowns with the formation of nanoscale holes; (4) the end of the anodization process (voltage shutdown).

layer, as well as replicas taken from it, respectively. The photo clearly shows the presence of nanoscale corrugation on the surface.

The chronoamperogram shown in Fig. 6 confirms the increase in the efficiency of the electrode material due to its nanostructuring. The applied voltage in all cases is 0.8 V (relative to the silver chloride electrode), the electrolyte is 0.1 M NaOH, and the electrode area is 1 cm^2 .

As shown in Fig. 6, the resulting nickel replicas provided a current return during oxidation of the model fuel (methanol) for fuel cells at 70–150 times greater amounts compared with smooth nickel. This result can be directly used to create improved and permanent sensors for methanol. To date, the reason for such a large increase in the efficiency of the electrodes in nanofilament remains unclear; whether it is a result of

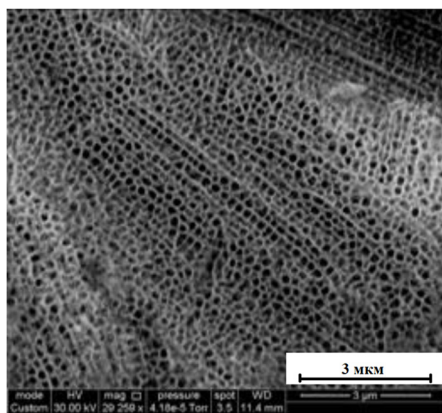


Fig. 4. An SEM image of an aluminum foil sample subjected to anodization at 70 V in 0.3 M phosphoric acid after removing the aluminum-oxide layer.

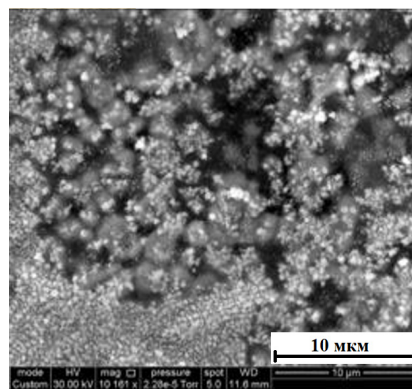


Fig. 5. An SEM image of a nickel replica sample from an aluminum foil subjected to anodization at 70 V in 0.3 M phosphoric acid after removing the aluminum-oxide layer.

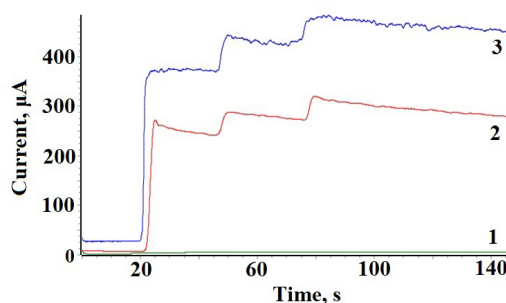


Fig. 6. Chronoamperograms when three 200- μL portions of methanol were sequentially added into an electrochemical cell with a volume of 100 mL: (1) compact nickel electrode; (2) nickel nanostructured material; (3) nickel nanostructured material with palladium nanoparticles.

an increase in the surface of the electrode or because geometric factors and a surface structure are also important. It is expected that in the future, the proposed approach will improve the performance of fuel cells and hydrogen-source electrolyzers.

CONCLUSIONS

The high-voltage anodic treatment of aluminum foil was used to form a nanoporated aluminum-oxide layer on a metal surface, and a nickel replica was formed on the surface of the treated aluminum. When testing the catalytic activity of the obtained replicas, using the electrochemical method on the example involving methanol oxidation, current densities were obtained at 70–150 times higher levels than on smooth nickel. Thus, it was found that the metal part of the anodized aluminum/aluminum-oxide-layer sandwich, despite having a much lower aspect ratio than the oxide part, was suitable as a template matrix for the creation of catalytically active materials and ultrafine electrodes made of nickel and other metals.

The results obtained herein can be used as a basis in chemical–technological installation aimed at creating initial (template) matrices for catalytically active materials.

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Authors' contribution

A.P. Antropov – formulation of the scientific concept, aims, and objectives of the study, processing and interpretation of experimental results;

N.K. Zaitsev – general management;

Y.D. Ryabkov – planning and conducting experiments;

N.A. Yashtulov – writing the manuscript, work group management;

P.N. Mudrakova – conducting experiments.

The authors declare no conflicts of interest.

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