

Nickel foam is widely used as a current lead/current collector and the basis of nickel hydroxide electrodes for various electrochemical devices – batteries, hybrid supercapacitors, devices for electrocatalytic oxidation of organic substances. The characteristics of commercial samples of nickel foam produced by Novomet-Perm (Russian Federation) obtained by electroless and then electrochemical nickel plating and Linyi Gelon LIB Co Ltd (China) obtained by electroless nickel plating were studied. The nature of passivity was determined by forming model samples of electrochemical and electroless nickel on a steel base. For the passive sample, activation was carried out by applying a layer of electrochemical nickel from an impact nickel plating solution. Activated, non-activated samples of nickel foam, as well as model samples, were studied by the methods of cyclic voltammetry and galvanostatic charge-discharge cycling in the supercapacitor mode. Comparative analysis of Chinese-made and Russian-made nickel foam samples showed significant passivity of the former – in cyclic voltammetry, the activity was 4.8 times lower, with galvanostatic charge-discharge cycling – 2.59 times lower. It was suggested that high passivity was determined by the fact that the sample consisted of Ni-P or Ni-B alloy. This assumption was proved by the method of natural simulation. The electrochemical activity of electroless nickel was 1.25 times lower than that of electrochemical nickel (according to cyclic voltammetry data) and 1.58 times lower (according to galvanostatic cycling data). For the first time, Chinese-made nickel foam (electroless nickel) was activated by applying a layer of electrochemical nickel from an impact nickel electrolyte. The high activation efficiency was shown as follows – on the cyclic curve, the specific current of the anodic peak increased 8.71 times, and with galvanostatic cycling, the increase in specific capacity was from 1.73 times (at $i=120 \text{ mA/cm}^2$) to 4.84 times (at $i=20 \text{ mA/cm}^2$)

Keywords: nickel foam, electrochemical nickel, electroless nickel, impact nickel, activation, current collector, supercapacitor

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COMPARATIVE INVESTIGATION OF DIFFERENT TYPES OF NICKEL FOAM SAMPLES FOR APPLICATION IN SUPERCAPACITORS AND OTHER ELECTROCHEMICAL DEVICES

Vadym Kovalenko

Corresponding author

PhD, Associate Professor

Department of Analytical Chemistry and Chemical Technology of Food Additives and Cosmetics*

Senior Researcher**

E-mail: vadimchem@gmail.com

Valerii Kotok

PhD, Associate Professor

Department of Processes, Apparatus and General Chemical Technology*

Senior Researcher**

*Ukrainian State University of Chemical Technology

Gagarina ave., 8, Dnipro, Ukraine, 49005

**Competence center “Ecological technologies and systems”

Vyatka State University

Moskovskaya str., 36, Kirov, Russian Federation, 610000

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1. Introduction

Supercapacitors (SC) are promising chemical current sources (CCS). SC are widely used to start various types of electric motors in electric vehicles, pumping stations, power tools, and other devices. SC can also be used as starters for gasoline and diesel engines, as uninterruptible power supplies for computers, medical devices, and even for entire buildings and premises. Supercapacitors are characterized by a high charge-discharge rate. As a result, at the Faraday electrode of the hybrid supercapacitor, the electrochemical process occurs on the surface and in the thin surface layer of the active substance particles. Therefore, special requirements are imposed on the active substance of such an electrode [1, 2]. The electrode, with $\text{Ni}(\text{OH})_2$ as the active substance, is widely used as the Faraday electrode of hybrid supercapacitors. Nickel hydroxide is used both independently [3], as a nanoscale [4]

or ultradispersed powder [5], and as a composite with nanocarbon materials (graphene oxide [6], carbon nanotubes [7]).

In supercapacitors, highly efficient current collectors must be used, which must ensure good electrical contact with the active substance. For this, two strategies are used: pasting the active substance onto the current collector [8, 9] and forming the active substance directly on the surface of the current collector [10, 11]. In addition to supercapacitors, pasted electrodes are widely used in high-power batteries [12, 13]. The production of an active substance on a current collector is used to form an electrode in electrocatalysis, for example, for oxygen evolution [14] or methanol electrolysis [15], as well as in electrochromic devices [16, 17].

When using an alkaline electrolyte, nickel foam is an effective current collector [8]. Nickel foam is produced by depositing nickel (electrolessly or electrochemically) on

graphitized polyurethane foam, followed by burning out the polymer. As a result, a highly porous open-cell system of nickel tubes is formed.

Particularly promising is the use of nickel foam as a current collector for a nickel oxide electrode and a current collector – the basis of a pasted nickel oxide electrode for electrocatalytic oxidation of organic substances [18]. This is because the nickel surface is covered with a thin layer of nickel oxide, which, in an alkaline medium, transforms into electrochemically active nickel hydroxide. Consequently, the capacity of the nickel oxide electrode in the supercapacitor mode will increase. The electrocatalytic activity of the electrode will increase as well.

However, the natural layer of nickel oxide on the surface of nickel foam is very thin and dense, its capacity and activity are negligible. In addition, nickel foam samples can be obtained by various methods, and the surface oxide layer can be highly passivated. At the same time, the activity of samples of nickel foam of various types and manufacturers is practically not studied, which complicates the production of electrochemical devices. Therefore, determining the surface activity of nickel foam and the ways of its activation is an urgent task.

2. Literature review and problem statement

There are two ways to activate nickel foam [19]:

- 1) the formation of an active layer of nickel hydroxide on the surface;
- 2) the increase in the specific surface area of nickel.

When implementing the first way, it is necessary to understand that the method and conditions of synthesis are directly determined by the electrochemical properties of nickel hydroxide. Nickel hydroxides can be obtained by chemical deposition by direct synthesis (adding an alkali solution to a nickel salt solution) [18] and reverse synthesis (adding a nickel salt solution to an alkali solution) [20, 21]. A two-stage high-temperature synthesis [22], a sol-gel method [23], and a homogeneous deposition method [24] are also used.

Electrochemical methods can also be used to obtain nickel hydroxide [25, 26], including synthesis in a slit diaphragm electrolyzer [27]. These methods make it possible to obtain electrochemically active samples of nickel hydroxide. However, these methods are of little use for activating the surface of nickel foam because $\text{Ni}(\text{OH})_2$ is formed as a separate phase. At the same time, to activate nickel foam, it is necessary to form a layer of nickel oxide or hydroxide directly on the surface.

Thin films of nickel oxide or hydroxide on the nickel surface can be obtained by chemical [28] and electrochemical [29, 30] methods. Electrochemically, $\text{Ni}(\text{OH})_2$ films can be obtained by cathodic deposition as a result of alkalization of the near-cathode layer of the solution [31]. A significant disadvantage of the method is low adhesion to the surface. A nickel hydroxide film can also be formed anodically [32, 33]. With anodic polarization, the nickel surface is oxidized with the formation of an oxide/hydroxide film [34, 35]. It should be noted that the films obtained by anodic oxidation have low porosity and thickness.

A nickel oxide film on a Ni surface is formed by a solvothermal process [36], thermal deposition [37, 38],

including the induction one [39], and thermal oxidation in the air [40].

It should be noted that the application of the oxide/hydroxide layer is only a part of the activation process. An etch surface development process can be used to improve adhesion and increase capacity. However, earlier in [19], a significant difference in the characteristics of nickel foam samples produced by different methods was revealed. For one of the samples, high passivity and low efficiency of activation both by etching (for surface development) and by forming a hydroxide layer were revealed.

3. The aim and objectives of the study

The study aims to determine the efficiency of various types of nickel foam as a current lead/current collector in various electrochemical devices, as well as to assess the possibility of activating the most passive samples. This will increase the performance of batteries, supercapacitors, and electrocatalytic oxidation devices.

To achieve the aim, the following objectives were set:

- to study the characteristics, including electrochemical ones, of nickel foam samples produced by electrochemical (RF) and electroless (China) nickel plating;
- to carry out simulation modeling of both types of nickel foam by applying electrochemical and electroless nickel on a steel base and study their electrochemical properties;
- to activate the passive sample of nickel foam by applying a layer of impact nickel and evaluate the activation effect.

4. Samples of nickel foam. Methods of activation and study of the characteristics of nickel hydroxide samples

Samples of nickel foam.

Two commercial samples of nickel foam were used for the study:

1) Nickel foam produced by *Novomet-Perm* (Russian Federation). Sample thickness – 2 mm. The sample of nickel foam was produced by applying a thin layer of electroless nickel, with further formation of a thicker Ni layer by electroplating. Sample marking – **NF-Ru**.

2) Nickel foam produced by *Linyi Gelon LIB Co Ltd* (China). Sample thickness – 0.3 mm. Presumably, the nickel foam sample was produced by applying a thin layer of electroless nickel. Sample marking – **NF-Ch**.

Methods for forming model samples.

To model samples of Russian and Chinese production, it was proposed to apply a layer of electrochemical (galvanic) and electroless nickel on a steel base:

- Sample with electrochemical nickel (marking – **E-Ni**).

Electrolyte composition:

- 1) $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ – 250–300 g/l;
- 2) $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ – 50–60 g/l;
- 3) H_3BO_3 – 25–40 g/l;
- 4) Ecomet H1A – 1.5–2.0 g/l;
- 5) Ecomet N1B – 7–10 ml/l.

Application mode: $i_c = 4\text{--}8 \text{ A/dm}^2$; $\text{pH} = 4.0\text{--}5.0$; $t = 55\text{--}60 \text{ }^\circ\text{C}$.

– Sample with electroless nickel (marking – **E-lessNi**).

Electrolyte composition:

1) $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ – 30–35 g/l;

2) $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ – 30–35 g/l;

3) $\text{Na}(\text{H}_2\text{PO}_2)$ – 15–20 g/l.

Application mode: $t=70-80^\circ\text{C}$, $\tau=2$ h.

Method for activating nickel foam samples.

The activation of passive samples of nickel foam was carried out by depositing a layer of impact nickel. **+E-ImpNi** was added to the sample marking.

Electrolyte composition:

– $\text{NiCl}_2 \cdot 5\text{H}_2\text{O}$ – 250 g/l;

– HCl – 50 g/l.

Application mode: holding time without current $\tau=3$ min; time under current $\tau=3$ min; $i=7$ A/dm².

Study of the characteristics of nickel foam samples.

The electrochemical properties of activated and non-activated samples of nickel foam, as well as model samples of electrochemical and electroless nickel, were studied by the methods of cyclic voltammetry and galvanostatic charge-discharge cycling. The samples were examined in an SES-2 cell (USSR) using an Ellins R-8 electronic potentiostat (RF). The samples under study were used as a working electrode. Electrolyte – 6M KOH. Counter electrode – Ni grid, reference electrode – saturated silver chloride. Cyclic voltammetry was carried out in the potential range from 0 to 500 mV (relative to the reference electrode), the sweep rate was 1 mV/s. Charge-discharge cycling in the supercapacitor mode was carried out at $i=20, 40, 80,$ and 120 mA/cm² (10 cycles each). The discharge curve was used to determine the discharge time. The specific capacities Q_{spec} (mAh/cm²) and F (F/cm²) were calculated from the magnitude of the discharge current and the geometric dimensions of the nickel foam sample.

5. Results of studying the characteristics of nickel foam samples

5.1. Comparative study of non-activated nickel foam samples

Comparative visual analysis of the **NF-Ru** (Russian-made) and **NF-Ch** (Chinese-made) samples revealed the following: the **NF-Ru** sample had a high thickness and high rigidity; the **NF-Ch** sample had a low thickness and high flexibility.

The results of studying the electrochemical activity of the samples are shown in Fig. 1, *a, b*. Cyclic voltammetry data confirmed the higher passivity of the **NF-Ch** foam sample (Chinese-made) compared to the sample of Russian **NF-Ru** nickel foam [19] – the specific currents of the cathode peak were 0.798 mAh/cm² and 3.47 mAh/cm².

Fig. 2, *a, b* shows the specific capacities of nickel foam samples obtained by cycling in the supercapacitor mode.

Charge-discharge cycling data confirmed cyclic voltammetry data about the lower activity of the **NF-Ch** sample compared to the **NF-Ru** sample. It should be noted that during a discharge up to $E=0$, the specific capacity of the **NF-Ru** sample did not practically change with an increase in the cycling current density, while the specific capacity of the **NF-Ch** sample increased with an increase in the current density.

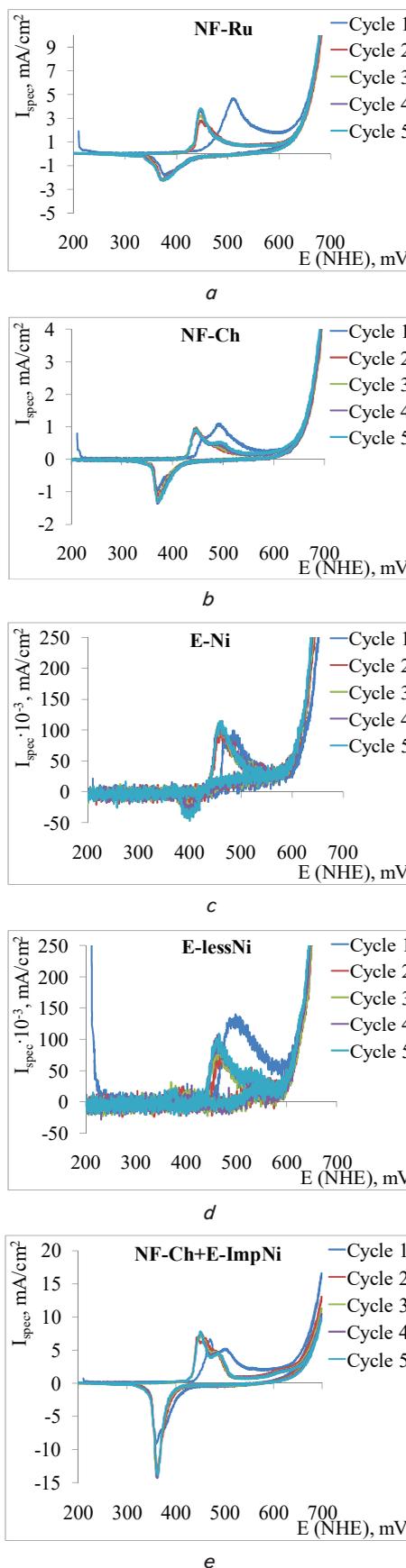


Fig. 1. Cyclic voltammograms of nickel foam samples: *a* – **NF-Ru**; *b* – **NF-Ch**; *c* – **E-Ni**; *d* – **E-lessNi**; *e* – **NF-Ch+E-ImpNi**

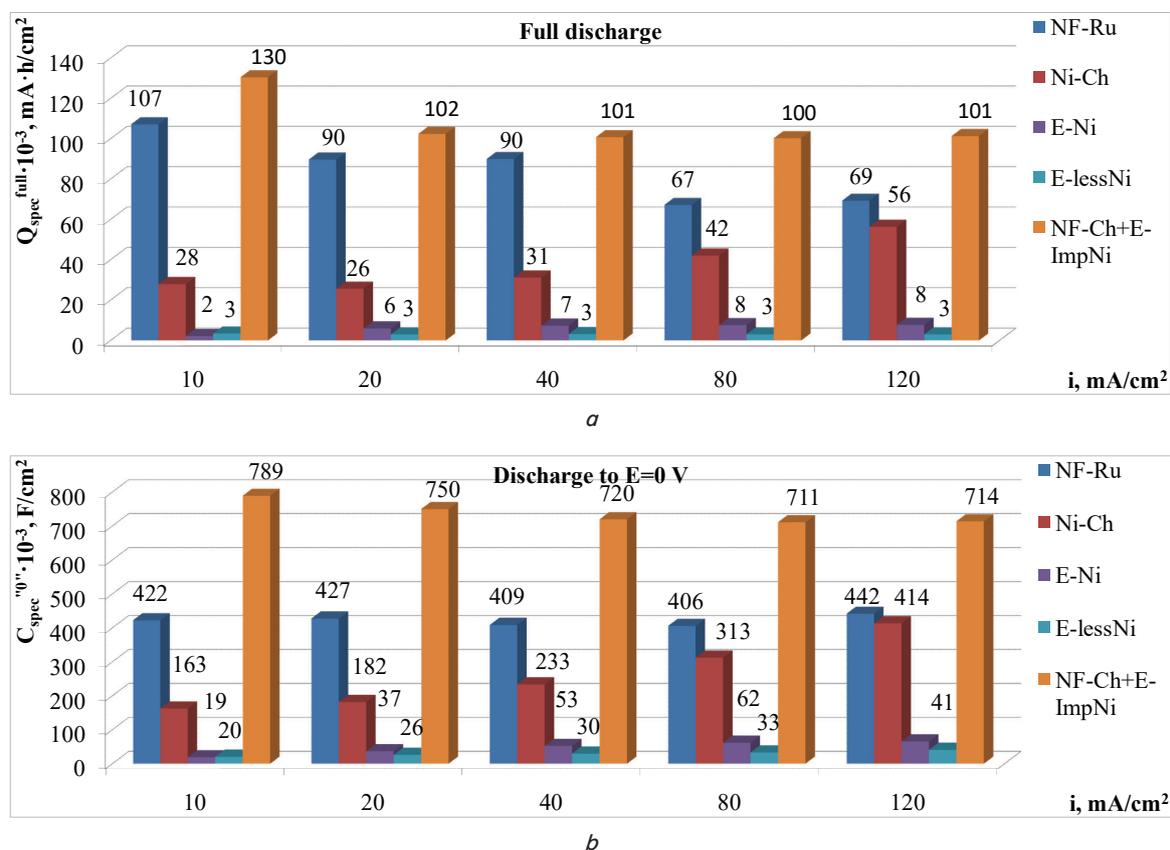


Fig. 2. Specific capacities of nickel foam samples: *a* – Q_{spec} , mA/h/cm², full discharge; *b* – C_{spec} , F/g; discharge up to $E=0$

5. 2. Study of the characteristics of model nickel samples

To confirm the hypothesis of passivity of Chinese-made nickel foam, model samples were obtained – 1) electrochemical (galvanic) nickel on a steel base; 2) electroless nickel on a steel base. Electroless nickel is a Ni-P alloy.

The electrochemical activity of the model samples studied by cyclic voltammetry is shown in Fig. 1, *c, d*. It is shown that electrochemical nickel (the **E-Ni** sample) had higher electrochemical activity than electroless nickel (the **E-lessNi** sample), which was indicated by the higher current of the anodic peak of the **E-Ni** sample. It should be noted that there were no cathodic (discharge) peaks on the cyclic curves of the **E-lessNi** sample; at the same time, for the **E-Ni** sample, clearly pronounced cathodic peaks with a specific current of 50 mA/cm² were observed on the cathodic curve.

The higher electrochemical activity of electrochemical nickel (the **E-Ni** sample) compared to electroless nickel (the **E-lessNi** sample) was also shown by the method of galvanostatic cycling when used in a supercapacitor (Fig. 2). The specific capacity at discharge up to $E=0$ (Fig. 2, *b*) for the **E-Ni** sample was 66 mF/cm², while, for the **E-lessNi** sample, it was 41 mF/cm².

5. 3. Study of the characteristics of the nickel foam sample activated by a deposited layer of impact nickel

The activation of the passive nickel foam made in China (the **NF-Ch** sample) was carried out by depositing a layer of nickel from an impact nickel plating electrolyte. The study of the activated **NF-Ch+E-ImpNi** sample by the method of cyclic voltammetry showed a significant increase in electrochemical activity (Fig. 1, *e*). The anodic curve of the activated **NF-Ch+E-ImpNi** sample showed two peaks with specific currents of 7.4 mA/cm² and 4.9 mA/cm² (compared

to 0.85 mA/cm² and 0.52 mA/cm² for the unactivated **NF-Ch** sample).

Charge-discharge cycling in the supercapacitor mode (Fig. 2) also showed a significant increase in the specific capacity of the activated **NF-Ch+E-ImpNi** sample: at $i=10$ mA/cm² 0.789 F/g (for the **NF-Ch** sample 0.163 F/g), at $i=120$ mA/cm² 0.714 F/g (for the **NF-Ch** sample 0.414 F/g).

6. Discussion of the results of a comparative study of the characteristics of non-activated and activated nickel foam samples and model samples

Based on the results of visual observation, it was concluded that the **NF-Ru** sample is most promising for use in stationary devices (batteries, supercapacitors, electrocatalysis devices). This sample of nickel foam can be used in a CCS with a flat design of electrodes, while the specific weight characteristics of these CCS will not be high due to the large weight of the current collector. At the same time, due to its low weight and high flexibility, the **NF-Ch** sample is recommended for use in mobile electrochemical devices of a roll design, in particular, supercapacitors, with high specific weight characteristics.

Cyclic voltammetry and galvanostatic charge-discharge cycling showed a lower activity of the **NF-Ch** nickel foam sample compared to the **NF-Ru** sample – the specific currents of the anodic peak were 0.85 mA/cm² and 3.47 mA/cm², respectively. In this case, the **NF-Ch** sample had 4.8 times less activity. During galvanostatic cycling, it was found that at $i=10$ mA/cm², the specific capacity for the **NF-Ru** sample was 422 F/g, and, for the **NF-Ch** sample, it was 2.59 times lower – 163 F/g. With an increase in the cycling current

density to 120 mA/cm², a significant difference was revealed in the behavior of the **NF-Ru** and **NF-Ch** samples. For the Russian-made sample (**NF-Ru**) obtained by the electrochemical method, the specific capacity increased slightly from 422 F/g to 442 F/g (+4.74 %). For the Chinese-made sample (**NF-Ch**), a more significant increase was revealed, from 163 mA/cm² to 414 mA/cm² (+153.99 %). This indicates significant processing of the oxide layer on the surface. It was suggested that this sample of nickel foam was produced by the electroless deposition of nickel. This formed a Ni-P or Ni-B alloy (depending on the reducing agent used). In this case, a denser and more passive oxide layer was formed on the surface.

To test this hypothesis, model samples were obtained by depositing layers of electrochemical nickel (the **E-Ni** sample as a model of the **NF-Ru** sample) and electroless nickel with a reducing agent – hypophosphite (the **E-lessNi** sample as a model of the **NF-Ch** sample). Cyclic voltammetry and charge-discharge cycling showed higher activity of electrochemical nickel (the **E-Ni** sample) in relation to electroless nickel (the **E-lessNi** sample) – the specific current of the anodic peak on the cyclic curve was 1.25 times higher (Fig. 1, *a, b*), and the specific capacity (at $i=120$ mA/cm²) was 1.58 times higher (Fig. 2, *a*). The absence of a cathodic peak on the cyclic curve of the **E-lessNi** sample should be noted. This fact clearly indicates the presence of an oxidizable component in the composition of nickel (in this case, P). All these data confirmed the assumption that the **NF-Ch** sample was produced by the method of electroless nickel plating and its passivity was associated precisely with the formation of a passive Ni-P or Ni-B alloy. However, it should be noted that the activity ratio of the **E-Ni/E-lessNi** model samples was lower than the ratio for the **NF-Ru/NF-Ch** samples. This is probably due to the fact that real nickel foam samples **NF-Ru** and **NF-Ch** were subjected to high-temperature heat treatment to burn out the polyurethane foam. In this case, the surface of electroless nickel was passivated.

There was an attempt to activate Chinese-made nickel foam (the **NF-Ch** sample) by depositing a nickel layer from an impact nickel plating electrolyte. The essence of impact nickel plating is as follows. The electrolyte contains a high concentration of hydrochloric acid. As a result, the current efficiency of nickel is 5–7 %, and 93–95 % of the current goes to hydrogen evolution. In this case, the H ad-atoms formed on the surface of the nickel foam reduce the passive oxide layer, and at the same time, the nickel layer is deposited. Thus, the passive oxide layer must be activated. The study of the electrochemical characteristics of the activated sample (**NF-Ch+E-ImpNi**) by the methods of cyclic voltam-

metry (Fig. 1, *e*) and galvanostatic charge-discharge cycling (Fig. 2) confirmed a sharp increase in activity compared to the non-activated **NF-Ch** sample – on the cyclic curve, the increase in the specific current of the first anodic peak was 8.71 times, and with galvanostatic cycling, the increase in the specific capacity was from 1.73 times (at $i=120$ mA/cm²) to 4.84 times (at $i=20$ mA/cm²).

It should be concluded that the surface activation of nickel foam is effective by depositing a nickel layer from an impact nickel plating electrolyte. The main limiting factor in this study was the limited range of samples – one of each type (obtained by electroless nickel plating and by electrochemical nickel plating). The conclusion about the effectiveness of surface activation by impact nickel plating should be checked on samples of other manufacturers' nickel foam.

7. Conclusions

1. A comparative study of the characteristics of the samples of nickel foam produced by *Novomet-Perm* (Russian Federation) and nickel foam produced by *Linyi Gelon LIB Co Ltd* (China) has been carried out. Russian-made nickel foam has a large thickness and rigidity and is recommended for use in stationary devices with flat-type electrodes. Chinese-made nickel foam has a low thickness and high flexibility and is recommended for use in mobile electrochemical devices (especially supercapacitors) with roll-type electrodes. It has been revealed that Chinese-made nickel foam (obtained by electroless nickel plating) has a lower activity than Russian-made nickel foam (obtained by electroless and then electrochemical nickel plating). According to the results of cyclic voltammetry, the specific current of the anodic peak is 4.8 times lower, according to galvanostatic charge-discharge cycling, the specific capacity is 2.59 times lower.

2. Reduced electrochemical activity of electroless nickel (Ni-P alloy) in comparison with electrochemical nickel has been proved by the method of simulation modeling. The electrochemical activity of electroless nickel is 1.25 times lower than that of electrochemical nickel (according to cyclic voltammetry data) and 1.58 times lower (according to galvanostatic cycling data).

3. Activation of Chinese-made nickel foam (electroless nickel) has been carried out by depositing a layer of electrochemical nickel from an impact nickel electrolyte. A high activation efficiency has been shown – on the cyclic curve, the specific current of the anodic peak increases 8.71 times; and with galvanostatic cycling, the increase in specific capacity is from 1.73 times (at $i=120$ mA/cm²) to 4.84 times (at $i=20$ mA/cm²).

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