

Nickel foam is widely used as a current lead/current collector and as the base of nickel hydroxide electrodes for hybrid supercapacitors. An investigation of the influence of activation conditions for a commercial sample of nickel foam produced by Linyi Gelon LIB Co Ltd (China) was carried out using the method of impact nickel plating. The morphology of activated and non-activated nickel foam samples was investigated by scanning electron microscopy. Activated and non-activated nickel foam samples were investigated by methods of cyclic voltammetry and galvanostatic charge-discharge cycling in the supercapacitor mode.

It was shown that upon activation at  $i=1 \text{ A/dm}^2$  and  $\tau=10 \text{ min}$ , a thin layer of porous nickel with incomplete coverage was formed. Activation with impact nickel at  $i=7 \text{ A/dm}^2$  and  $\tau=3 \text{ min}$  revealed the formation of a nickel coating with a highly developed surface, on which local cracks were found as a result of the accumulation of internal stresses. Activation with impact nickel at  $i=1 \text{ A/dm}^2$  and  $\tau=10 \text{ min}$  led to the formation of a coating with a highly developed surface, with significant peeling of the coating.

Cyclic voltammetry showed high efficiency of impact nickel activation at  $i=7 \text{ A/dm}^2$ ,  $\tau=3 \text{ min}$ , and  $i=20 \text{ A/dm}^2$ ,  $\tau=5 \text{ min}$ . The specific current of the cathode peak increased 6.06–6.44 times with respect to the non-activated sample. The investigation of the activated samples' electrochemical characteristics by the galvanostatic cycling method showed that impact nickel activation at  $i=1 \text{ A/dm}^2$  and  $\tau=10 \text{ min}$  was insufficient. It was found that at a discharge up to  $E=0 \text{ V}$ , the maximum specific capacitance of  $0.731 \text{ F/cm}^2$  was obtained for samples activated by impact nickel at  $i=7 \text{ A/dm}^2$  and  $\tau=3 \text{ min}$ . The increase in specific capacitance compared to the non-activated sample was 4.49 times. At full discharge, the highest electrochemical activity was found for nickel foam samples activated by impact nickel at  $i=20 \text{ A/dm}^2$  and  $\tau=5 \text{ min}$ . The specific capacitance was  $0.505 \text{ mA}\cdot\text{h/cm}^2$ , and it increased 9.02 times

**Keywords:** nickel foam, impact nickel, activation, current collector, supercapacitor, specific capacitance, cyclic voltammetry, charge-discharge cycling, surface development

# ACTIVATION OF NICKEL FOAM, AS A CURRENT COLLECTOR OF A SUPERCAPACITOR, BY IMPACT NICKEL PLATING: INFLUENCE OF TREATMENT CONDITIONS

**Vadym Kovalenko**

Corresponding author

PhD, Associate Professor

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

E-mail: vadimchem@gmail.com

**Valerii Kotok**

PhD, Associate Professor

Department of Processes, Apparatus and General Chemical Technology\*

**Volodymyr Verbitskiy**

Doctor of Pedagogical Sciences, Professor, Director

Department of Medical, Biological

and Valeological Basics of Life and Health Protection

National Pedagogical Dragomanov University

Pyrogova str., 9, Kyiv, Ukraine, 01601

National Ecology and Nature Center,

Vyshgorods'ka str., 19, Kyiv, Ukraine, 04074

**Volodymyr Medianyuk**

PhD, Associate Professor

Department of Mining Engineering and Education

Dnipro University of Technology

Dmytra Yavornytskoho ave., 19, Dnipro, Ukraine, 49005

\*Ukrainian State University of Chemical Technology

Gagarina ave., 8, Dnipro, Ukraine, 49005

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## 1. Introduction

Supercapacitors (SC) are promising chemical power sources (CPS). SCs are widely used to start various types of electric motors in electric vehicles, pumping stations, power tools, and other devices, particularly in mining [1]. SCs can also be used as starters for gasoline and diesel engines, and as uninterruptible power supplies for computers, medical devices, and even entire buildings and premises. Supercapacitors are characterized by a high charge-discharge rate. As a

result, on the Faraday electrode of the hybrid supercapacitor, the electrochemical process proceeds on the surface and in a thin surface layer of active substance particles. Therefore, special requirements are imposed on the active substance of such an electrode [2, 3]. 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 [4], in the form of a nanosized [5] or ultrafine powder [6], and as a composite with nanocarbon materials (graphene oxide [7], carbon nanotubes [8]).

In supercapacitors, high-efficiency current leads should be used, which should provide good electrical contact with the active substance. For this, two strategies are used: pasting the active substance on the current collector [9, 10] and forming the active substance directly on the surface of the current collector [11, 12]. In addition to supercapacitors, pasted electrodes are widely used in high-power batteries [13, 14]. The production of an active substance on a current collector is used to form an electrode in electrocatalysis, for example, for oxygen evolution [15] or methanol electrolysis [16], as well as in electrochromic devices [17, 18].

When using an alkaline electrolyte, the most used material is Ni [19]. The most efficient current collector is nickel foam [9, 20]. Nickel foam is produced by coating nickel (chemically or electrochemically) to graphitized polyurethane foam, followed by polymer burning. 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 lead, which is the basis of a coated nickel oxide electrode for electrocatalytic oxidation of organic substances [21]. This is because the surface of nickel is covered with a thin layer of nickel oxide, which transforms into electrochemically active nickel hydroxide in an alkaline medium. And, consequently, the capacitance of the nickel oxide electrode in the supercapacitor mode will increase. The electrocatalytic activity of the electrode also increases.

However, the natural layer of nickel oxide on the surface of nickel foam is very thin and dense, and its capacitance and activity are negligible. In addition, nickel foam samples can be obtained by various methods, and the surface of the oxide layer can be highly passivated. This significantly reduces the efficiency of the nickel-foam-based electrode. Therefore, it is an urgent task to determine effective methods for activating the nickel foam surface and the conditions for carrying out the activation.

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## 2. Literature review and problem statement

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Two ways can be used to activate nickel foam [22]:

- 1) formation of an active layer of nickel hydroxide on the surface;
- 2) an 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 directly determine 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) [21] and reverse synthesis (adding a nickel salt solution to an alkali solution) [23, 24]. The two-stage high-temperature synthesis [25], the sol-gel method [26], and the homogeneous deposition method [27] are also used.

Electrochemical methods can be used to obtain them [28, 29], including synthesis in a slit diaphragm electrolyzer [30]. These methods make it possible to obtain electrochemically active samples of nickel hydroxide. However, these methods are of little use for activating the nickel foam surface 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 nickel oxide or hydroxide layer directly on the surface.

Thin films of nickel oxide or hydroxide on the nickel surface can be obtained by chemical [31] and electrochemical [32, 33] methods. The chemical method can be used

to obtain porous films of pure  $\text{Ni}(\text{OH})_2$  [34] and films of Ni-Mn layered double hydroxide [35]. A hydroxide film can be obtained with preliminary etching [36].  $\text{Ni}(\text{OH})_2$  films can be obtained electrochemically by cathode deposition as a result of the alkalization of the near-cathode layer of the solution [37, 38]. A significant disadvantage of the method is low adhesion to the surface. A nickel hydroxide film can also be formed anodically [39, 40]. During anodic polarization, the nickel surface is oxidized to form an oxide/hydroxide film [41, 42]. It should be noted that the films obtained by anodic oxidation have a low thickness and porosity.

A nickel oxide film on the Ni surface is formed by a solvothermal process [43], thermal deposition [44, 45], including induction [46], and thermal oxidation in the air [47]. Microwave processing is also used to obtain an oxide film [48].

The second way is to remove the passive layer and increase the specific surface area. The removal of the passive layer and an increase in the specific surface area can be carried out by etching, similar to the selective processing of alloys [49]. In [50, 51], nickel foam was treated in an HCl solution using different acid concentrations [52]. However, the authors of those publications talked about cleaning the surface of nickel foam, probably referring to the removal of the active layer. However, it is more correct to speak about activation. Another way to increase the surface is the deposition of highly developed coatings with nickel or its alloys [53]. However, the disadvantage of this method is the need for careful surface preparation. The work [22] showed the high passivity of thin and flexible samples of nickel foam, which were an alloy of Ni-P and Ni-B. For such samples, high passivity and low activation efficiency were revealed both by etching (for surface development) and by forming a hydroxide layer.

In [54], the high activation efficiency of this type of nickel foam by the method of impact nickel coating was shown. However, in this work, impact nickel was coated only at one current density and coating time; the effect of coating conditions was not investigated. It should be concluded that for a promising method of nickel foam activation by impact nickel plating, the effect of current density and coating time has not been investigated.

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## 3. The aim and objectives of the study

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The work aims to determine the effect of conditions for impact nickel coating on the efficiency of activation of nickel foam as a current collector of chemical power sources. This will increase the characteristics of supercapacitors.

To achieve the aim, the following objectives were set:

- to carry out activation of nickel foam samples by impact nickel coating at different current densities and coating times, and investigate the surface morphology of nickel foam;
- to investigate the electrochemical characteristics of activated samples of nickel foam as a supercapacitor current collector.

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## 4. Materials and methods

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### 4.1. Object and hypothesis of the study

The object of the study is the technology of nickel foam activation. The hypothesis during the investigation is that the activation efficiency depends on the conditions of impact nickel coating, namely the current density and coating time.

#### 4. 2. Nickel foam sample

A commercial sample of nickel foam produced by Linyi Gelon LIB Co Ltd (China) is used for the study. This type of nickel foam is produced by thin-layer electroless nickel plating. Sample marking is NF-Ch.

#### 4. 3. Activation method for nickel foam samples

Activation of passive nickel foam samples is carried out by coating a layer of impact nickel [54].

Electrolyte composition:

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

–  $\text{HCl}$  – 50 g/l.

Holding time without current  $\tau=3$  min.

Coating conditions:

–  $i=1$  A/dm<sup>2</sup>,  $\tau=10$  min. Sample marking NF-Ch-imp-1-10;

–  $i=7$  A/dm<sup>2</sup>,  $\tau=3$  min. Sample marking NF-Ch-imp-7-3;

–  $i=20$  A/dm<sup>2</sup>,  $\tau=5$  min. Sample marking NF-Ch-imp-20-5.

#### 4. 4. Investigation of the nickel foam samples' characteristics

The surface morphology of the nickel foam samples is investigated by scanning electron microscopy (SEM) using a 106-I microscope (SELM, Ukraine).

The electrochemical properties of activated and non-activated nickel foam samples are studied by cyclic voltammetry and galvanostatic charge-discharge cycling methods described in [54]. The samples are investigated using a special YaSE-2 cell (USSR) and an Ellins R-8 electronic galvanostat/potentiostat (RF). The investigated samples are used as the working electrode. Electrolyte – 6M KOH. A Ni grid is used as a counter electrode, and a saturated silver chloride electrode is used as a reference electrode. During cyclic voltammetry, a potential sweep is carried out in the range from 0 to 500 mV (relative to the reference electrode) at a rate of 1 mV/s. Charge-discharge cycling in the supercapacitor mode is carried out at current densities of 20, 40, 80, and 120 mA/cm<sup>2</sup> (10 cycles each). The discharge time is determined from the discharge curve, and the specific capacitance  $Q_{sp}$  (mA·h/cm<sup>2</sup>) and  $F$  (F/cm<sup>2</sup>) are calculated from the discharge current and overall geometrical dimensions of the nickel foam sample.

### 5. Results of investigation of the nickel foam samples' characteristics

#### 5. 1. Results of investigation of the nickel foam samples' morphology

SEM images of activated and non-activated nickel foam samples are shown in Fig. 1. A non-activated sample of NF-Ch (Fig. 1, *a–c*) is a metal filament, on the surface of which scale-like structures with a diameter of about 5  $\mu\text{m}$  are distinguished.

The SEM images of the sample activated with impact nickel at 1 A/dm<sup>2</sup> and 10 minutes of coating (sample NF-Ch-imp-1-10) at low magnifications (Fig. 1, *d, e*) show no fundamental difference from the non-activated sample. However, at  $\times 5000$  magnification, it can be seen that the foam nickel filaments are covered with a thin layer of porous nickel, with the presence of cavities. Activation of nickel foam at 7 A/dm<sup>2</sup> and 3 minutes of coating (sample NF-Ch-imp-7-3) leads to the formation of a thicker nickel layer, and the flaky texture of the base is not visible (Fig. 1, *g, h*). The nickel layer is finely porous, cracks in some places, and is covered with fine powder-like

particles. Activation of nickel foam at 20 A/dm<sup>2</sup> and 5 minutes of coating (sample NF-Ch-imp-20-5) leads to the formation of a nickel layer, and the scaly texture of the base is not visible (Fig. 1, *g, h*). The nickel precipitate is finely porous, cracks in some places, and is covered with fine powder-like particles.

Fig. 2 shows SEM images of the NF-Ch-imp-7-3 sample after galvanostatic cycling.

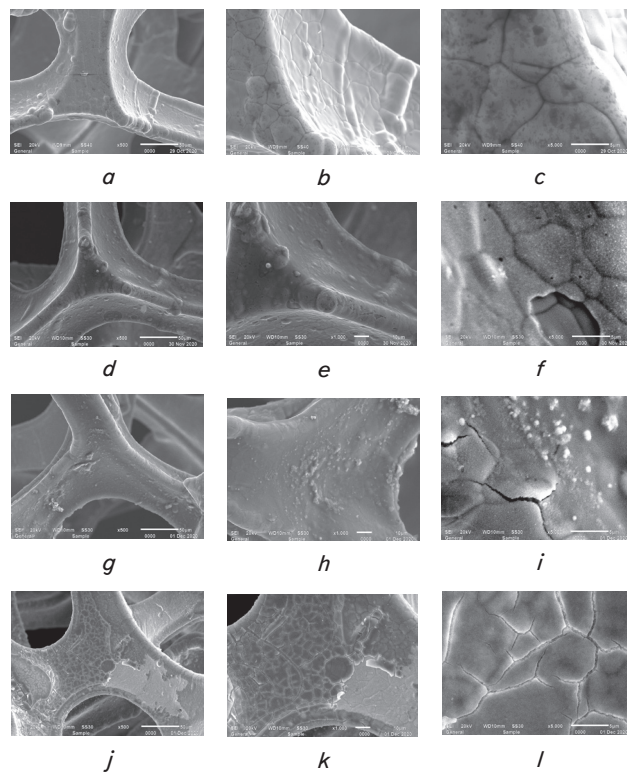


Fig. 1. SEM images of nickel foam samples: *a, b, c* – NF-Ru; *d, e, f* – NF-Ch-imp-1-10; *g, h, i* – NF-Ch-imp-7-3; *j, k, l* – NF-Ch-imp-20-5. Magnification: *a, d, g, j* –  $\times 500$ ; *b, e, h, k* –  $\times 1000$ ; *c, f, i, l* –  $\times 5000$

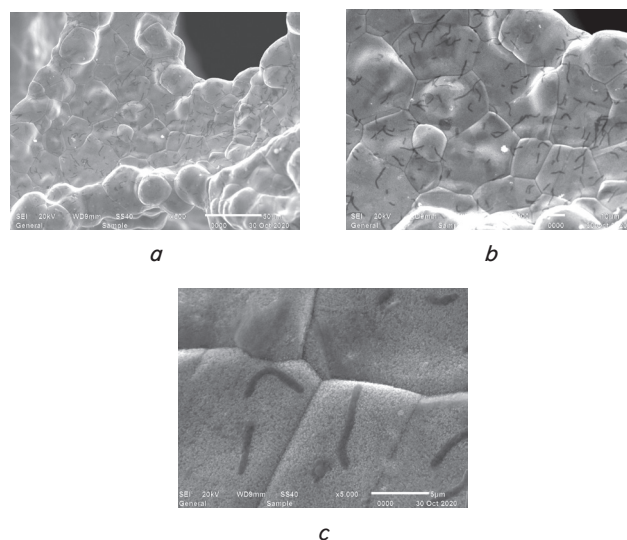


Fig. 2. SEM images of the NF-Ch-imp-7-3 nickel foam sample after galvanostatic cycling. Magnification: *a* –  $\times 500$ ; *b* –  $\times 1000$ ; *c* –  $\times 5000$

After charge-discharge cycling, a highly porous layer on the surface is clearly visible (Fig. 2, *c*) with darker worm-like



stripes. At the same time, it should be noted that the scaly texture is revealed after electrochemical processing.

### 5. 2. Results of investigation of the nickel foam samples' electrochemical characteristics

Fig. 3 shows cyclic voltammograms of non-activated and activated nickel foam samples. The voltammogram of the non-activated NF-Ch sample (Fig. 3, *a*) reveals low electrochemical activity – the specific peak of the cathode peak was 0.92 A/cm<sup>2</sup>. Treatment by impact nickel coating leads to a significant activation of foam nickel – the specific currents of the cathode peak in the series of samples “NF-Ch-imp-1-10 – NF-Ch-imp-7-3 – NF-Ch-imp-20-5” are 4.8, 8 and 8.5 A/cm<sup>2</sup>, respectively. It should also be noted that for the non-activated NF-Ch sample, the potential of the first anodic peak is 495–500 mV. At the same time, for all activated samples, the potential of the first anodic peak is 450–465 mV.

Fig. 4, *a, b* show the specific capacitance of nickel foam samples obtained by cycling in the supercapacitor mode. The charge-discharge cycling data confirms the cyclic voltammetry data of the lower activity of the non-activated NF-Ch sample. The impact nickel coating leads to the activation of foam nickel with an increase in specific capacitance. It should be noted that at a discharge up to  $E=0$  (Fig. 4, *b*), the specific capacitance increases in the series of samples “NF-Ch-imp-1-10 – NF-Ch-imp-20-5 – NF-Ch-imp-7-3”. With an increase in the cycling current density, the specific capacitance of the activated samples decreases, while the specific capacitance of the NF-Ch sample increases with an increase in the current density. The maximum specific capacitance of 0.731 F/cm<sup>2</sup> is obtained for the NF-Ch-imp-7-3 sample (at  $i=10$  mA/cm<sup>2</sup>), the increase in specific capacitance compared to the non-activated sample is 4.49 times. At the same time, at full discharge, the specific characteristics of the activated NF-Ch-imp-1-10 sample increase insignificantly compared to the non-activated NF-Ch sample.

For the activated sample NF-Ch-imp-7-3 (under impact nickel coating conditions of 7 A/dm<sup>2</sup> and 3 min),

the maximum specific capacitance of 0.199 mA·h/cm<sup>2</sup> is obtained at a cycling current density of 40 mA/cm<sup>2</sup>. The highest electrochemical activity is found for the sample NF-Ch-imp-20-5 – the specific capacitance is 0.505 mA·h/cm<sup>2</sup> (at a cycling current density of 120 mA/cm<sup>2</sup>), the increase in specific capacitance compared to the non-activated sample is 9.02 times.

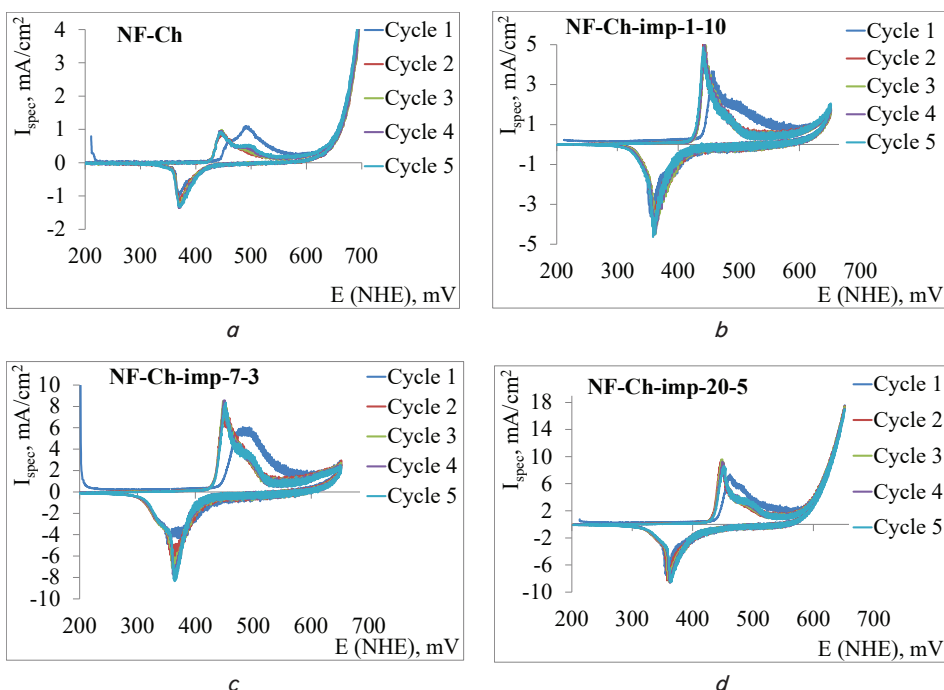


Fig. 3. Cyclic voltammograms of nickel foam samples: *a* – NF-Ch; *b* – NF-Ch-imp-1-10; *c* – NF-Ch-imp-7-3; *d* – NF-Ch-imp-20-5

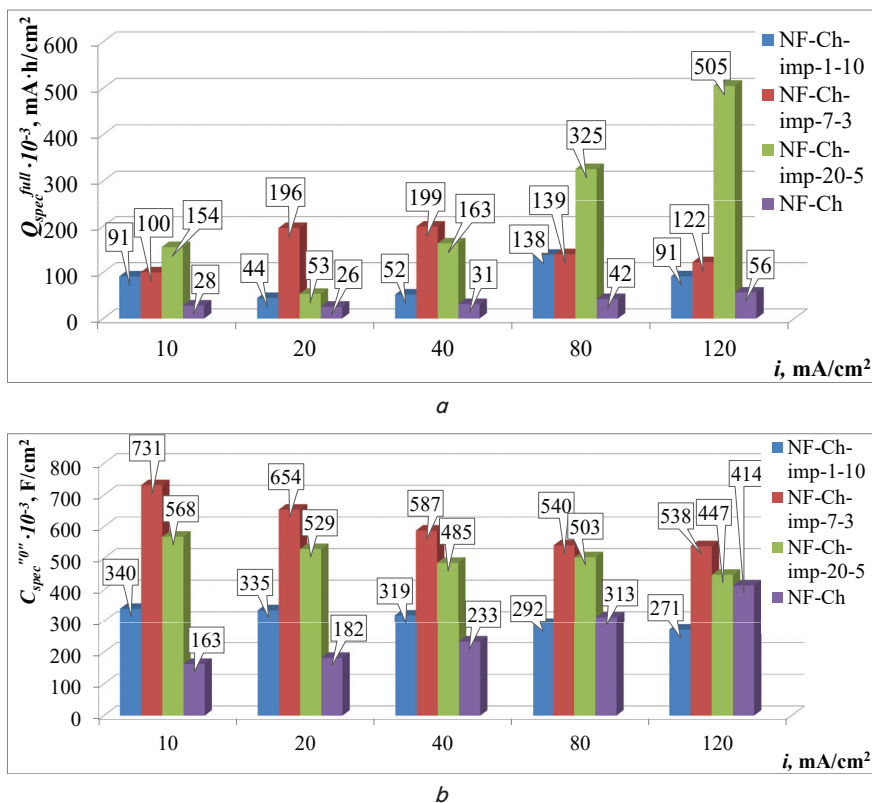


Fig. 4. Specific capacitance of nickel foam samples: *a* –  $Q_{sp}$ , mA·h/cm<sup>2</sup>, full discharge; *b* –  $C_{sp}$ , F/g; discharge to  $E=0$

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

When discussing the results of studying the influence of the activation conditions on the characteristics of foam nickel samples, it is necessary to consider the mechanism of activation by the method of impact nickel coating. Electrolyte for impact nickel plating contains a high concentration of HCl. Therefore, during nickel plating, depending on the current density, the current efficiency of Ni is 2–7 %, and 93–98 % of the current goes to hydrogen evolution. In this case, the H ad-atoms formed on the nickel surface foam reduce the passive oxide layer (this mechanism is known as hydrogen reduction at the moment of release). At the same time, a certain amount of nickel metal is coated. In this case, the coating of nickel occurs at current densities above the limiting one, and the metal deposits in the form of dendrites with a highly developed surface. In this way, the passive surface of the nickel foam is activated.

Influence of impact nickel coating conditions on nickel foam surface morphology.

Activation at a low current density (1 A/dm<sup>2</sup>) and a coating time of 10 min (NF-Ch-imp-1-10 sample) leads to the formation of a thin nickel layer on the nickel foam surface (Fig. 1, *d–f*). The coating thickness is less than 2 μm because the coating reproduces the scaly morphology of the nickel foam base (Fig. 1, *d*). The nickel layer contains micropores, and the coating is partially incomplete, with gaps (Fig. 1, *f*). Most likely, the activation effect of this coating will be negligible. Activation at a current density of 7 A/dm<sup>2</sup> and a coating time of 3 min (sample NF-Ch-imp-7-3) leads to the formation of a thicker nickel layer (Fig. 1, *g*), which also has micropores and partially cracks (Fig. 1, *j*). The surface of the coating contains powder formations. Such a morphology of the coating is explained by the fact that with an increase in the current density, a more porous, loose, and dendritic layer is formed. In this case, there is a significant increase in the specific surface area. However, an increase in internal stress in the precipitate is observed, which leads to partial cracking and peeling. A further increase in the activation current density to 20 A/dm<sup>2</sup> (sample NF-Ch-imp-20-5) leads to the coating of a highly porous active nickel coating (Fig. 1, *j–l*). However, this coating is highly stressed, which leads to cracking and even significant peeling (Fig. 1, *k, l*) and even separation of nickel coated. Despite the high surface, this coating may have a lower activity due to the possible separation of the layer part during operation.

Influence of impact nickel coating conditions on the electrochemical characteristics of activated samples.

The investigation of the electrochemical characteristics of the activated samples by cyclic voltammetry (Fig. 3) and galvanostatic charge-discharge cycling (Fig. 4) confirmed a sharp increase in activity compared to the non-activated NF-Ch sample. On the cyclic curves in the series of samples “NF-Ch-imp-1-10 – NF-Ch-imp-7-3 – NF-Ch-imp-20-5”, the values of the specific currents (A/dm<sup>2</sup>) of the cathode and anode peaks are “4.7 – 8.0 – 8.5” and “4.88 – 8.35 – 9.24”, respectively. The increase in the values of the specific current of the cathode peak in the same series of activated samples concerning the specific current of the non-activated sample is the following series “356 % – 606 % – 644 %”. These data indicate a significantly higher activation efficiency at 7 A/dm<sup>2</sup> and 3 min coating time, as well as at 20 A/dm<sup>2</sup> and 10 min coating time. These data are confirmed by the SEM results.

The results of galvanostatic charge-discharge cycling (Fig. 3) confirm insufficient activation by impact nickel at 1 A/dm<sup>2</sup> and a coating time of 10 min (NF-Ch-imp-1-10 sample). At a discharge up to  $E=0$  V, the specific capacitance of the NF-Ch-imp-1-10 sample at high cycling current densities (80 mA/cm<sup>2</sup> and 120 mA/cm<sup>2</sup>) turns out to be lower than the specific capacitance of the non-activated NF-Ch sample (Fig. 4, *b*). At full discharge, higher specific capacitance is characteristic of the NF-Ch-imp-20-5 sample. For this sample, as the current density increases from 20 A/dm<sup>2</sup> to 120 A/dm<sup>2</sup>, the specific capacitance sharply increases from 53 mA·h/cm<sup>2</sup> to 505 mA·h/cm<sup>2</sup>. The high electrochemical activity (and high activation efficiency) is due to the formation of an impact nickel layer with a highly developed surface upon activation. However, at a discharge up to  $E=0$  V (Fig. 4, *a*), the specific capacitance of the NF-Ch-imp-20-5 sample activated with impact nickel at 20 A/dm<sup>2</sup> and the coating time of 5 min turns out to be lower than the specific capacitance of the NF-Ch-imp-7-3 sample. For the NF-Ch-imp-20-5 sample, the specific capacitance is 0.568 F/cm<sup>2</sup> (at  $i=20$  mA/cm<sup>2</sup>) and 0.414 F/cm<sup>2</sup> (at  $i=120$  mA/cm<sup>2</sup>). At the same time, as for the NF-Ch-imp-7-3 sample, the specific capacitance is 0.731 F/cm<sup>2</sup> (at  $i=20$  mA/cm<sup>2</sup>) and 0.538 F/cm<sup>2</sup> (at  $i=120$  mA/cm<sup>2</sup>). It should be noted that both samples are characterized by a decrease in specific capacitance with an increase in the cycling current density. The cycling data at full discharge are probably due not only to the factor of increasing the specific surface area of impact nickel but also to the presence of internal stresses in the coat and the possibility of the film peeling off the base due to them. Activation with impact nickel at 20 A/dm<sup>2</sup> and a coating time of 5 min (NF-Ch-imp-20-5 sample) leads to the formation of a highly developed nickel layer, which is prone to peeling (Fig. 1, *l*). As a result, at full discharge, the specific capacitance decreases precisely because of the peeling of impact nickel from the nickel foam base.

It should be concluded that the activation of the nickel foam surface is effective by coating a nickel layer from an electrolyte of impact nickel plating at current densities of 7–20 A/dm<sup>2</sup> and a coating time of 3–5 min. The main limiting factor of this investigation is the limited range of conditions for the activation of nickel foam samples by impact nickel. To eliminate this factor, it is necessary to investigate the effect of current density and coating time separately. In addition, the conclusion made about the conditions for effective surface activation by impact nickel plating should be verified on samples of nickel foam from other manufacturers. A limitation of the study is that the electrochemical characteristics of the activated nickel foam base itself have been investigated. As a result, the data can be used in special types of supercapacitors and electrocatalysis devices. For use in supercapacitors (general form), it is necessary to prepare samples of a pasted oxy-nickel electrode with a nickel-foam current collector activated by impact nickel under various conditions and investigate their electrochemical characteristics.

## 7. Conclusions

1. Activation of nickel foam produced by Linyi Gelon LIB Co Ltd (China) has been carried out by impact nickel plating under various conditions. Scanning electron microscopy has shown that, upon activation at 1 A/dm<sup>2</sup> and a coating time of 10 min, a thin layer of porous nickel is formed with a partial non-coverage of the substrate. Upon activation

with impact nickel at  $7 \text{ A/dm}^2$  and a coating time of 3 min, the formation of a nickel coating with a highly developed surface has been revealed, which has local cracks as a result of the accumulation of internal stresses. Activation with impact nickel at  $20 \text{ A/dm}^2$  and a coating time of 5 min has resulted in significant peeling of the coating.

2. The electrochemical characteristics of nickel foam samples activated under different conditions have been investigated by the methods of cyclic voltammetry and galvanostatic charge-discharge cycling. It has been shown that activation with impact nickel at  $1 \text{ A/dm}^2$  and a coating time of 10 min is insufficient. It has been found that at a discharge up to  $E=0 \text{ V}$ , the maximum specific capacitance of  $0.731 \text{ F/cm}^2$  is obtained for a nickel foam sample activated with impact nickel at  $7 \text{ A/dm}^2$  and a coating time of 10 min.

The increase in specific capacitance compared to the non-activated sample is 4.49 times. At full discharge, the highest electrochemical activity is found for a nickel foam sample activated with impact nickel at  $20 \text{ A/dm}^2$  and a coating time of 5 min. The specific capacitance is  $0.505 \text{ mA}\cdot\text{h/cm}^2$ , and the increase in specific capacitance compared to the non-activated sample is 9.02 times.

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### Conflict of interest

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The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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