

Nanostructured electrode material for energy storage

Anna O. Diteleva¹, Dmitry Yu. Kukushkin¹, Vladimir V. Sleptsov¹

1 Moscow Aviation Institute, 4 Volokolamskoe Highway, Moscow 125993, Russian Federation

Corresponding author: Anna O. Diteleva (anna.diteleva@mail.ru)

Received 24 February 2025 ♦ Accepted 28 April 2025 ♦ Published 2 October 2025

Citation: Diteleva AO, Kukushkin DY, Sleptsov VV (2025) Nanostructured electrode material for energy storage. *Modern Electronic Materials* 11(3): 155–162. <https://doi.org/10.3897/j.moem.11.3.143304>

Abstract

This work is devoted to the development of an electrode material for energy storage devices and its nanostructuring technology, which significantly improve energy characteristics. The electrode material consists of a conductive carbon flexible matrix with a high specific surface area filled with silver nanoparticles measuring 10–40 nm. The design and manufacturing technology of a supercapacitor based on an aqueous electrolyte with an operating voltage of 2.6 V and a specific energy consumption of 5.5 Wh/kg are presented.

Keywords

supercapacitor, energy storage, nanostructuring, water electrolyte, nanoparticles, carbon matrix, carbon fiber

1. Introduction

In recent years, energy storage technologies have become increasingly relevant in light of the growing need for efficient and reliable energy sources. Supercapacitors stand out from other devices due to their ability to provide high power density and long service life. However, in order to achieve optimal characteristics of supercapacitors, it is necessary to improve the properties of their electrode materials. The main parameters for promising energy storage are the increase in energy intensity and safety. To do this, it is necessary to use various electrodes with a significantly higher theoretical capacity, increase the voltage limit and switch to safe electrolytes.

Nanostructuring of the electrode material is one of the promising areas that contribute to improving the characteristics of energy storage devices. Nanostructures such as nanostructured porous carbon materials, metals and metal oxides can significantly increase the surface area and improve electrical conductivity, which in turn leads

to an increase in the capacity and speed of charging/discharging devices.

Another factor that improves the efficiency of energy storage devices is the manufacturing technology. Existing thick-film technologies realize only 10–15 % of the theoretically possible energy intensity. It is promising to use a thin-film technology for manufacturing energy storage devices, which makes it possible to reduce the internal resistance of cells by orders of magnitude, which will lead to a decrease in heat generation during cell operation and, accordingly, to an increase in specific energy consumption and operational safety, which are the most important requirements for cells.

In the near future, the 3rd generation of energy storage devices with a high content of Li and Ni, which are used as a cathode, have the greatest development potential (Fig. 1). The anode is carbon in the form of a highly porous matrix containing nanoparticles of Si, Ge, Sn, P, and Sb materials. An elastic graphene-based matrix with Si nanoparticles (Figs 2a and 2b) has a specific anode energy consumption of ~2000 mAh/g, which is twice

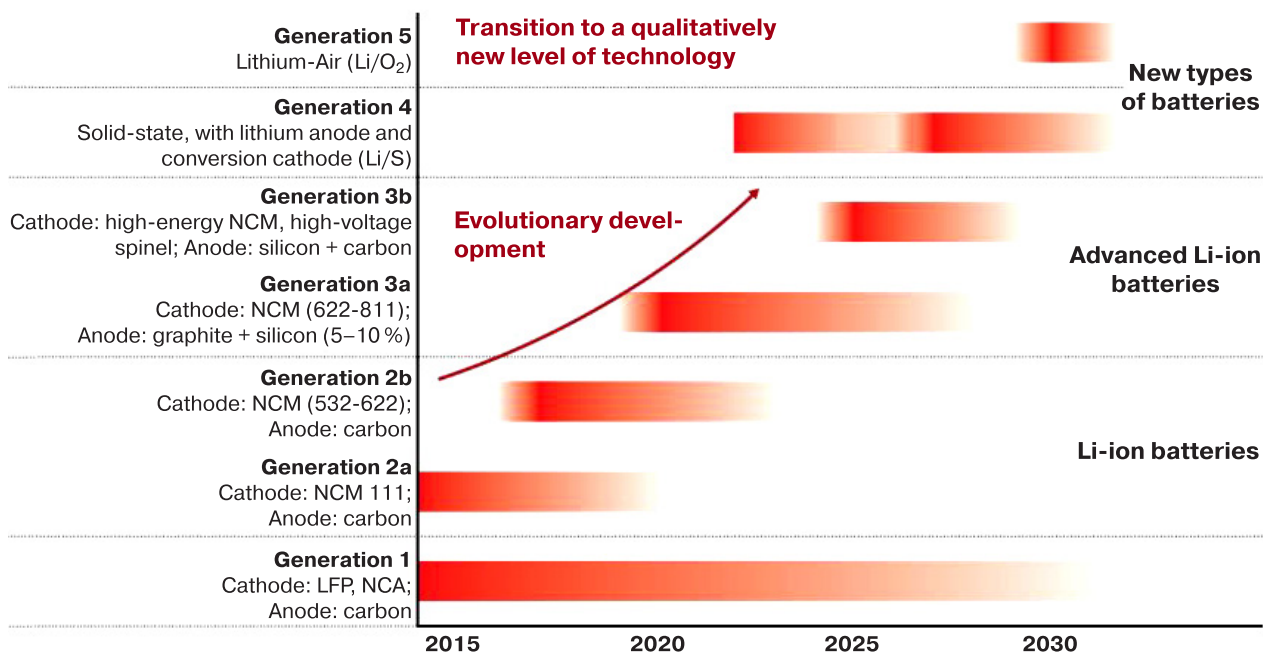


Figure 1. Efficiency of energy storage devices by generation

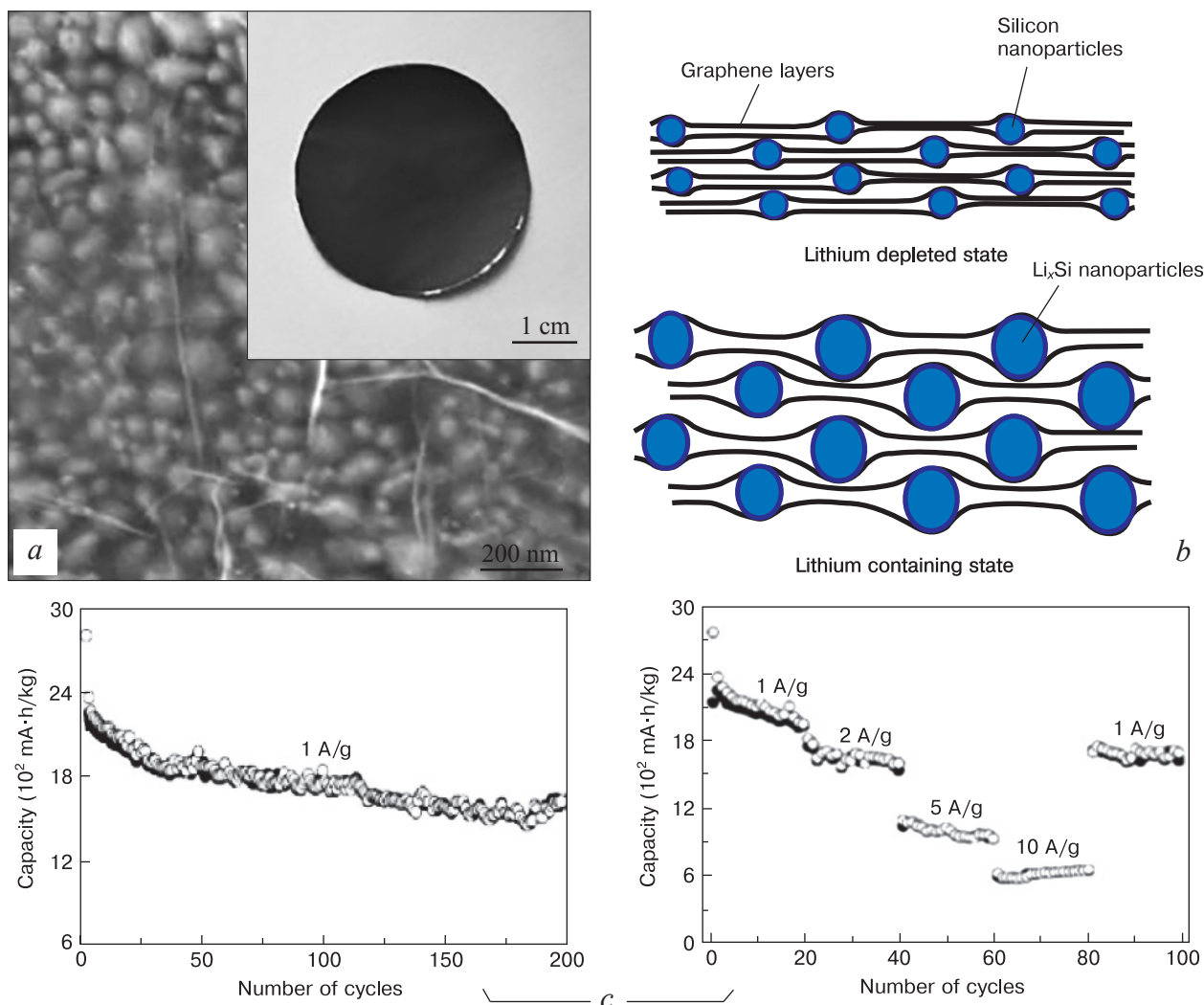


Figure 2. Micrograph (a), the appearance of an anode made of an elastic graphene-based matrix with Si nanoparticles (b) and diagrams of the dependence of the specific capacitance on the cycle number (c)

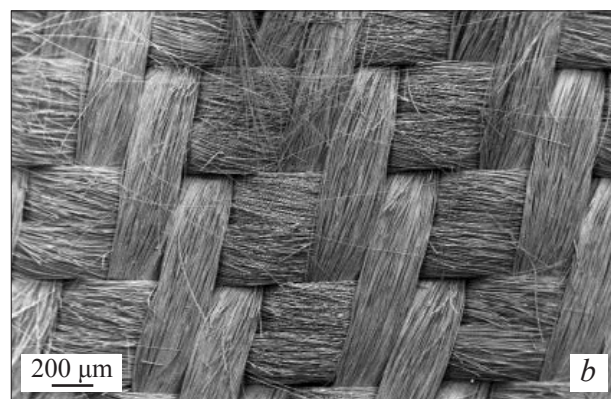
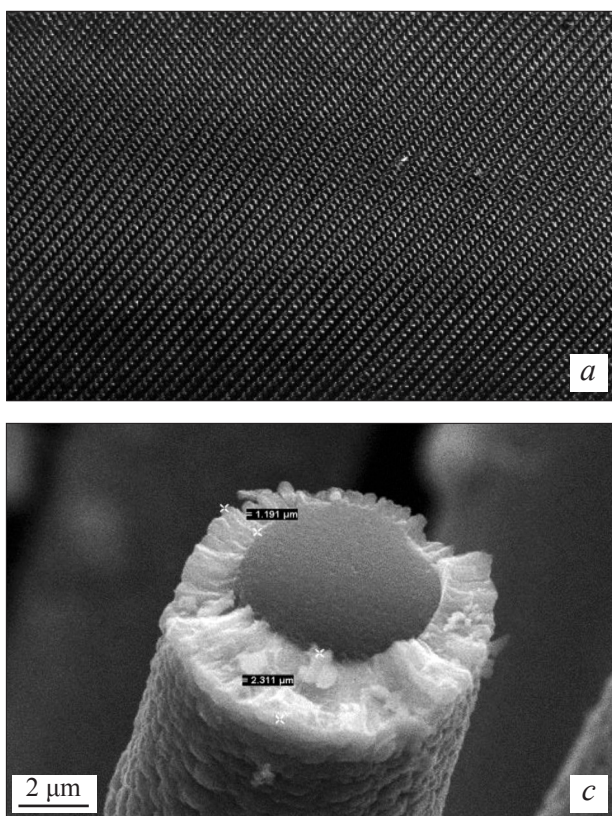


Figure 3. Photo of the initial fabric of the Busofit type: (a) the initial carbon material Busofit; (b) the carbon material Busofit coated with titanium; (c) a single thread of the fabric of the carbon material Busofit coated with titanium (2,311 μm)

the capacity of graphene (1000 mA·h/g) and its stability also increases with an increase in the number of cycles (Fig. 2c). The main disadvantage is the relatively low electrical potential (no more than 1 V) [1–6].

The next important task is to increase the security of the drive. The electrothermal safety of devices based on aqueous electrolytes is higher in comparison with storage devices with organic electrolytes, which is very important during their production and operation. Also, the manufacture of such devices is associated with less technical difficulties, because a special atmosphere is not needed, and at the same time production costs are reduced.

However, carbon capacitors have a higher energy and power density in organic electrolytes compared to aqueous ones due to the high operating voltage, the maximum operating voltage is limited by the “window” of electrochemical stability, which theoretically cannot exceed 1.23 V. A way to increase the energy density of a device with aqueous electrolytes is to overcome the “window” of electrochemical stability of water.

The creation of electrode materials with a highly developed surface increases the speed of electrochemical processes in terms of the geometric contact surface of the electrode material – electrolyte, which leads to a decrease in charging time and heat dissipation. By forming the nanostructure of an electrode material, its energy, electrical and operational characteristics can be changed. The presence of a highly developed surface increases the rate of electrochemical reactions, which can lead to a decrease in charging time. Promising materials for filling the carbon matrix are Li and its alloys, Si, Al, Na, Sn, Mg, Zn, Ni, Co, Ag, etc.

The aim of this work was to develop a promising electrode material for energy storage devices and a manufacturing technology that would significantly improve the energy characteristics.

2. Methods

The developed electrode material consists of a highly porous carbon conductive matrix filled with metal nanoparticles. A carbon fabric of the Busofit type with a high specific surface area (1200 m^2/g), shown in Fig. 1a, was used as the starting material.

The metallization of the carbon matrix took place in a vacuum using magnetron technology on a roll-up unit of the UMRM-1 type. Magnetron sputtering provides good adhesion of the deposited layers, the possibility of deposition of multilayer coatings, large sizes of processed samples, the absence of high temperatures on the treated surface, a droplet component and a low rate of spraying of the material, which allows you to control the process of film growth and obtain a wide range of coating thicknesses – from fractions to micrometer units. A layer of titanium with a thickness of 2 μm was applied on one side of the carbon material and 8 μm on the other. The sprayed titanium layer made it possible to reduce the high internal resistance of the initial fiber Busofit [7–9].

Figure 3 shows photos of the surface of the initial carbon material of the Busofit type without coating, and in Fig. 3b – with a layer of titanium applied to the surface of the Busofit type material and a separate thread of the



Figure 4. Electric pulse plant for the production and deposition of metal nanoparticles (*a*) and its reactor (*b*)

fabric of the carbon material Busofit with a layer of titanium ($2.311 \mu\text{m}$) in Fig. 3c.

Promising materials for filling the carbon matrix are Li and its alloys, Si, Al, Na, Sn, Mg, Zn, Ti, Ni, Co, Ag, etc. [5–10]. A distinctive feature of the production technology of such capacitor structures is the ability to work with materials with a highly developed surface (more than $1000 \text{ m}^2/\text{g}$). Therefore, thin-film technologies have significantly wider possibilities in comparison with traditional thick-film technologies in creating promising designs of electrochemical systems.

Nanostructuring was performed on the above-described porous carbon fiber Busofit with a thin layer of titanium. Silver nanoparticles with a size of 10–40 nm were used as nanoparticles [11, 12]. Nanostructuring was carried out in an electric pulse installation, which allows simultaneously obtaining nanoparticles of various metals and depositing them inside the material.

Figure 4 shows an electric pulse unit for the production of metal nanoparticles (Fig. 4a) and its reactor (Fig. 4b). The mechanism for producing nanoparticles is based on an electrohydraulic shock, which consists in the formation of a gas bubble when a pulsed voltage (4–10 kV)

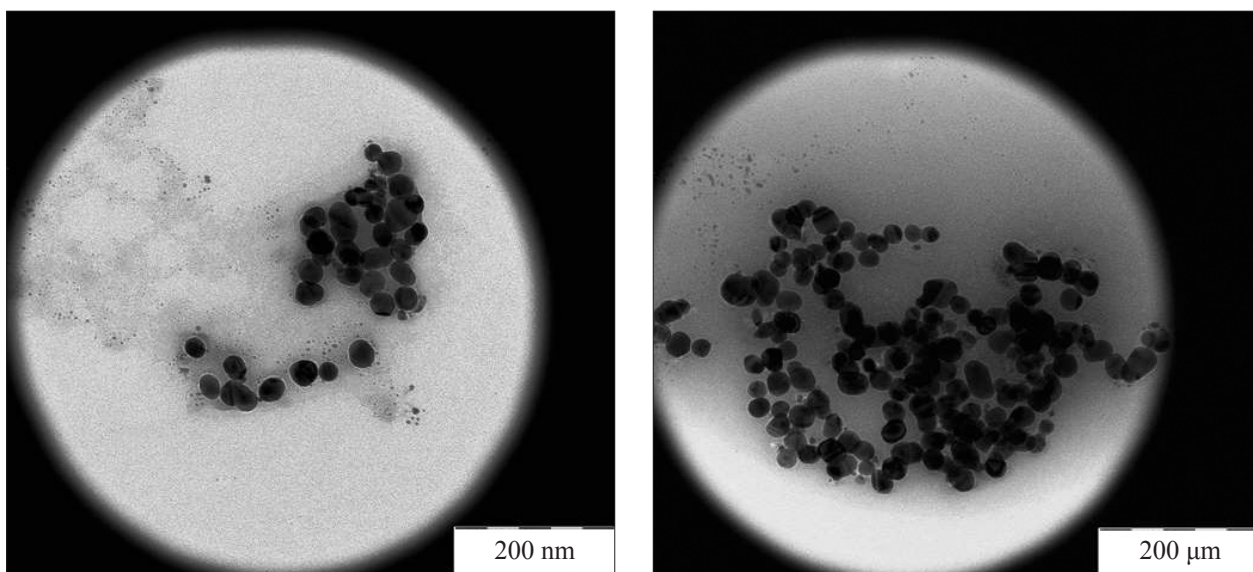


Figure 5. Images of the obtained silver nanoparticles on an electron microscope



Figure 6. Accessories for fastening the processed material Busofit in an electric pulse installation

is applied between electrodes made of the material necessary to produce nanoparticles, an electric pulse discharge with the release of metal nanoparticles into a liquid (distilled water) and subsequent collapse of the gas bubble. The result is a solution containing nanoparticles of the corresponding metal in distilled water.

The electric pulse installation allows to obtain and apply nanoparticles of various metals and alloys, which

is achieved by changing the electrodes of the installation to other electrodes made of the required metal and alloy. Figure 5 shows the obtained silver nanoparticles with a size of 10–40 nm.

The electric pulse unit allows not only to obtain nanoparticles in a liquid medium, but also simultaneously to position them into the processed material using additional equipment to which the processed material is attached (Fig. 6).

3. Results

Using an electric pulse unit, silver nanoparticles were deposited into the carbon metallized titanium fiber Busofit, shown in Fig. 7. Deposition of zinc nanoparticles into the carbon fiber Busofit made it possible to obtain a coating with a high specific surface area (Fig. 8).

When magnesium nanoparticles were deposited by this method on the surface of the Busofit material, it was possible to obtain a thin film (Fig. 9).

X-ray spectral microanalysis of the carbon tissue of Busofit with silver nanoparticles deposited by electric pulse technology allows us to conclude that there are no foreign impurities in the nanostructuring process (Fig. 10).

Studies have been conducted on the effect of silver nanostructuring on the energy, electrical and operational characteristics of energy storage devices. For this purpose, identical laboratory samples of two types of supercapacitors were made. In the first type, titanium-metallized carbon material Busofite without nanostructuring was used as an electrode material, and in the second type, titanium-metallized carbon material Busofite nanostructured with silver.

The design of such supercapacitors is layered and consists of laying electrode materials, a separator made of capacitor paper between them and two current leads

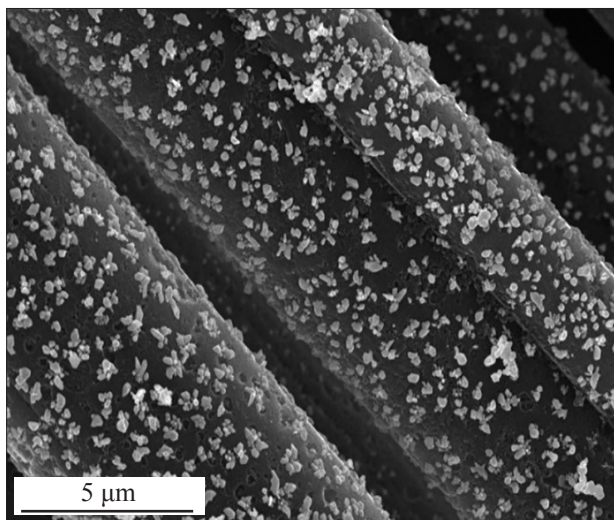


Figure 7. Carbon material Busofit with silver coating using electric pulse technology

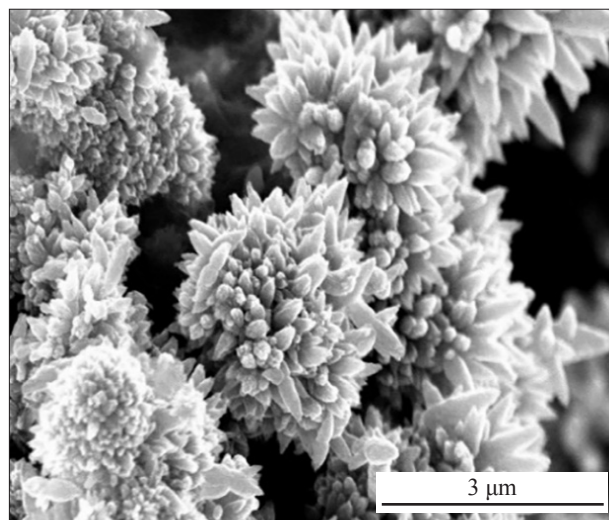


Figure 8. Metallization of Busophyte on an electric pulse installation with zinc nanoparticles

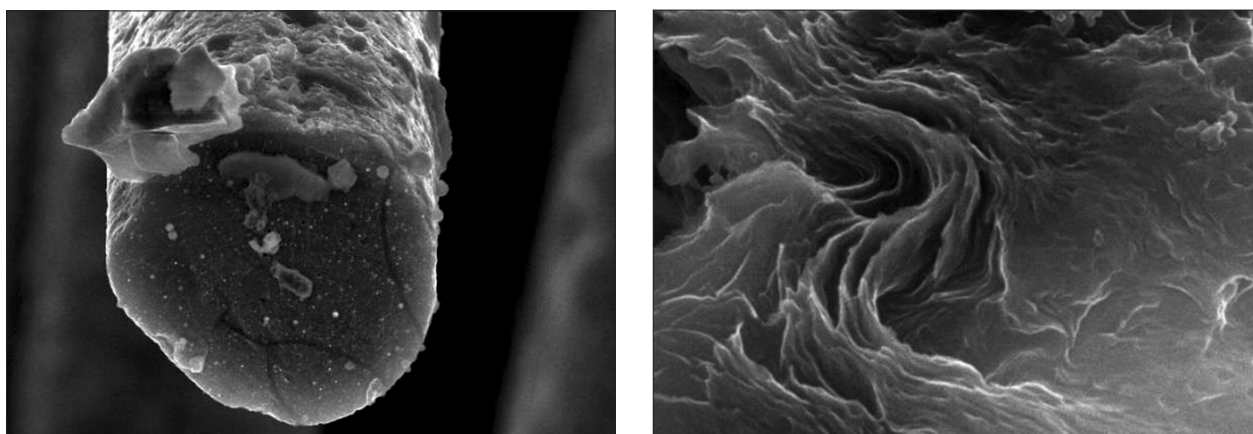


Figure 9. Metallization of Busophyte on an electric pulse installation with magnesium nanoparticles

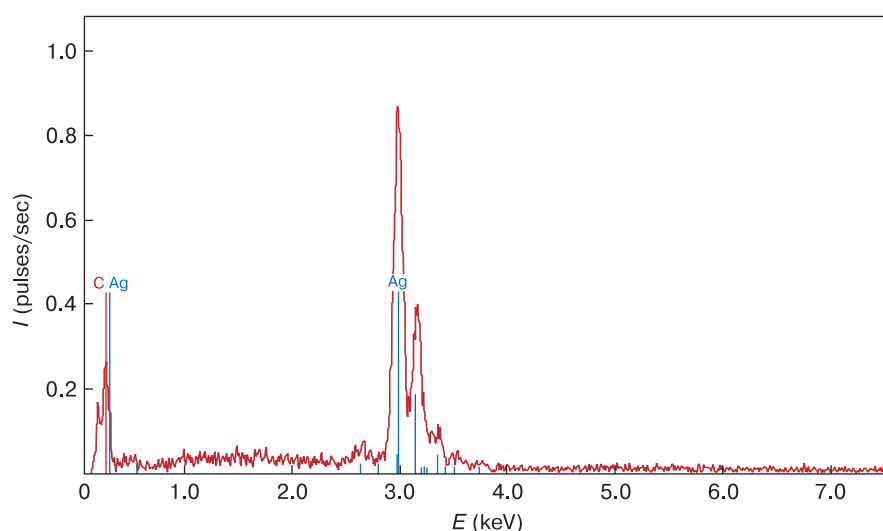


Figure 10. X-ray spectral microanalysis of carbon tissue Busofit with silver nanoparticles using electric pulse technology

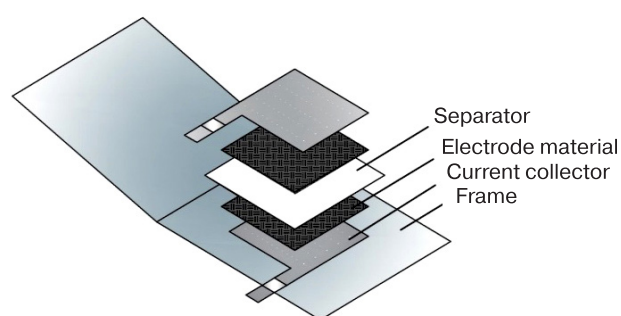


Figure 11. Manufactured capacitors and design

made of titanium foil. Figure 11a shows manufactured laboratory samples of supercapacitors and a single-layer structure in Fig. 11b.

An aqueous 10 % NaCl solution was used as the electrolyte. Titanium foil with a thickness of 50 μm was used

for current collectors. The samples were assembled outdoors at room temperature.

Figures 12 and 13 show the charge/discharge curves of supercapacitors without nanostructuring (see Fig. 12) and nanostructured with silver using electric pulse

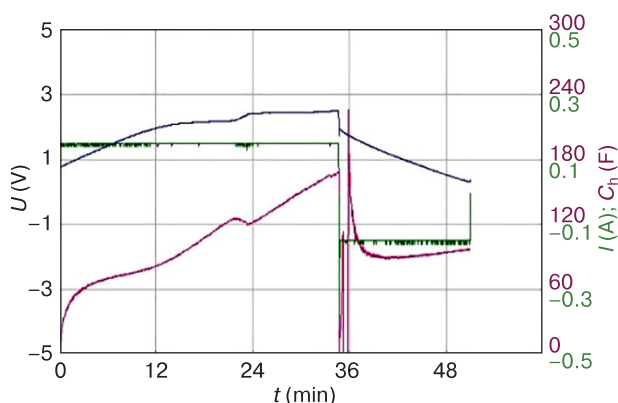


Figure 12. Charge/discharge curves of supercapacitors on an aqueous electrolyte with a single-layer structure

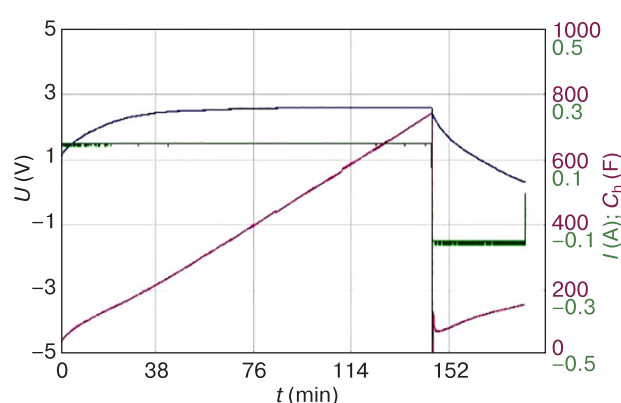


Figure 13. Charge/discharge curves of silver nanostructured supercapacitors on an aqueous electrolyte with a single-layer structure

Table 1. Comparison of characteristics of water condensers based on carbon materials

Carbon material	Electrolyte	Specific capacity/ specific energy consumption	Maximum voltage (V)
Activated carbon	10% NaCl	19 $\mu\text{mF}/\text{cm}^2$	1.2
Graphite powder	10% NaCl	35 $\mu\text{mF}/\text{cm}^2$	1.2
Graphite cloth	0.168 N NaCl	10.7 $\mu\text{mF}/\text{cm}^2$	1.2
Carbon cloth	5–10% Li_2SO_4	4.3 F/g	1.6
Carbon cloth	5–10% Na_2SO_4	3.8 F/g	1.6
Carbon cloth	5–10% K_2SO_4	3.6 F/g	1.6
Our sample	10% NaCl	1.64 F/ cm^2 ; 5.92 F/g; 5.56 Wh/kg	2.6

technology (see Fig. 13). It can be seen from the charge/discharge curves that supercapacitors nanostructured with silver have a longer discharge time and capacity and a lower internal resistance. Both types of supercapacitors on an aqueous electrolyte withstood a voltage of 2.5 V.

Characteristics of an aqueous electrolyte supercapacitor without nanostructuring:

- specific energy consumption – 4.2 Wh/kg;
- internal resistance of the ESR – 1.47 Ohm;
- discharge time – 15 min;
- operating voltage – 2.5 V.

Characteristics of a silver nanostructured supercapacitor using electric pulse technology, on an aqueous electrolyte:

- specific energy consumption – 5.56 Wh/kg;
- the internal resistance of the ESR – 0.435 Ohm;
- the discharge time – 35 min;
- the operating voltage – 2.6 V.

Laboratory samples of supercapacitors, nanostructured with silver, made on an aqueous electrolyte, withstood a voltage of 2.6 V without electrolyte decomposition processes. The specific energy intensity was 5.56 Wh/kg with a sample weight of 25 g. Nano-structuring of electrode materials with silver using electric pulse technology

allowed to increase the discharge time by 2 times, reduce the internal resistance of the ESR by 70 % and increase the specific energy intensity by 32 %.

4. Discussion

A comparison was made with similar capacitors made on the basis of aqueous electrolytes and carbon materials. On average, the specific capacity for supercapacitors on an aqueous electrolyte does not exceed 35 $\mu\text{mF}/\text{cm}^2$, 3.8 F/g and 1.6 V. The manufactured sample with the developed electrode material exceeds analogues in specific capacity by 37 %, and in operating voltage by 62 %.

Conclusion

A model, design and technology of an electrode material and a supercapacitor based on an aqueous electrolyte have been developed. The electrode material is a conductive carbon flexible matrix with a high specific surface area, nanostructured with silver nanoparticles 10–40 nm in size. The possibility of overcoming the “window” of the electrochemical potential of decomposition of water (1.23 V) in a capacitor with an aqueous electrolyte is shown. The operating voltage of the manufactured cells is 2.6 V. The operating voltage and specific capacity of the manufactured silver nanostructured samples using electric pulse technology are higher than those of analogues.

The developed electric pulse technology makes it possible to apply nanostructures of other metals and alloys and has a high prospect also in the development of cathode materials of chemical current sources.

Acknowledgments

The work was carried out within the framework of the state task of the Ministry of Education and Science of the Russian Federation, topic No. FSFF-2023-0008.

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