

Original article

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INFLUENCE OF TEMPERATURE ON THERMOELECTRIC EFFECT IN THE COMPOSITE MATERIAL BASED ON CARBON NANOTUBES AND POLYANILINE

A. A. Tretyakov¹, V. M. Kapralova¹, V. V. Loboda¹,
I. Yu. Sapurina², N.T. Sudar¹✉

¹ Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russia;

² Institute of Macromolecular Compounds RAS, St. Petersburg, Russia

✉ sudar53@mail.ru

Abstract. In the paper, the effect of temperature (range 22–140 °C) on the power factor (PF) of composite material made of multi-walled carbon nanotubes with a 10 nm thick polyaniline coating applied to their surface has been studied. The conductivity value of the coating was varied by treating the composite with buffer solutions of different pH values. The nanotubes were randomly oriented relative to each other in the composite samples under study. It was found that the composite material where polyaniline was in an oxidized conductive form had the highest PF value. In that sample, the PF values were about 0.5 $\mu\text{W}/(\text{m}\cdot\text{K}^2)$ at room temperature and about 1 $\mu\text{W}/(\text{m}\cdot\text{K}^2)$ at 140 °C.

Keywords: thermoelectricity, polyaniline, nanotube, Seebeck coefficient, conductivity, buffer solution, pH value

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ВЛИЯНИЕ ТЕМПЕРАТУРЫ НА ТЕРМОЭЛЕКТРИЧЕСКИЙ ЭФФЕКТ В КОМПОЗИЦИОННОМ МАТЕРИАЛЕ НА ОСНОВЕ УГЛЕРОДНЫХ НАНОТРУБОК И ПОЛИАНИЛИНА

А. А. Третьяков¹, В. М. Капралова¹, В. В. Лобода¹,
И. Ю. Сапурина², Н. Т. Сударь¹✉

¹ Санкт-Петербургский политехнический университет Петра Великого,
Санкт-Петербург, Россия;

² Институт высокомолекулярных соединений РАН, Санкт-Петербург, Россия
✉ sudar53@mail.ru

Аннотация. В работе исследовано влияние температуры (диапазон 140 – 22 °С) на коэффициент мощности (КМ) композиционного материала из многостенных углеродных нанотрубок с нанесенным на их поверхность полианилиновым покрытием (толщина около 10 нм). Величина проводимости покрытия варьировалась путем обработки композита буферными растворами с различными значениями рН. В исследуемых образцах композита нанотрубки были беспорядочно ориентированы друг относительно друга. Установлено, что наибольшим значением КМ обладает композиционный материал, в котором полианилин находится в окисленной проводящей форме. У этого образца при комнатной температуре значение КМ составило примерно 0,5 мкВт/(м·К²), а при 140 °С – около 1 мкВт/(м·К²).

Ключевые слова: термоэлектричество, полианилин, нанотрубка, коэффициент Зеебека, проводимость, буферный раствор, водородный показатель

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Introduction

Interest towards clean energy has grown over the recent years. Thermoelectric generators (TEG), capable of converting thermal energy directly into electrical energy, show promise as sources of such energy [1–4].

Inorganic semiconductor materials are commonly used to fabricate TEG. For example, highly doped lead chalcogenides PbTe, PbSe, PbS are the key materials for applications in thermoelectrics [4, 5]. The efficiency of energy conversion by thermoelectrics can be estimated by the power factor P , defined as $P = \alpha^2 \sigma$ (α is the Seebeck coefficient, σ is the electrical conductivity). Power factor values amounting to about 400 $\mu\text{W}/(\text{m}\cdot\text{K}^2)$ have been achieved in the temperature range from 600 to 950 K for inorganic thermoelectrics. However, along with such an advantage as high energy conversion efficiency, inorganic semiconducting materials also have a number of



disadvantages: they are considerably expensive and difficult to produce, highly toxic, and therefore cannot be used in everyday life. The negative properties of such thermoelectrics significantly limit their potential applications as TEG.

Novel non-toxic materials can be proposed as an alternative to traditional inorganic thermoelectric materials, in particular, electroconductive polyconjugated polymers. They have a range of unique properties, namely: low specific gravity, low thermal conductivity, easy synthesis, small cost, as well as, importantly, potential for constructing flexible TEG [3]. Remarkably, organic TEG exhibit acceptable thermoelectric characteristics over the temperature range of 200–380 K, i.e., are capable of operating at much lower temperatures than their inorganic counterparts. Therefore, they can be installed in residential buildings and even included in the composition of electronic textiles for clothing [6].

On the other hand, despite these advantages, organic electrically conductive polymers have a relatively low conductivity and a low Seebeck coefficient, so they cannot compete on an equal footing with inorganic thermoelectric materials [7].

Composites with organic and inorganic components can offer far better thermoelectric properties; composite materials based on conductive polymers and carbon nanotubes (CNTs) have been the subject of particular attention [8–11]. The properties of such composites depend on a variety of factors, including the type (single or multilayer), chirality, purity, defect density, and size (length and diameter) of the nanotubes, dispersion state and alignment of the nanotubes in the polymer matrix, and interfacial adhesion between the nanotube and the polymer matrix. These factors should be taken into account when presenting, interpreting and comparing the results obtained for nanotube composites with polymers [12].

Another study [10] examined a sample of polyaniline composite (PANI) with multilayer (multi-walled) carbon nanotubes (MWCNTs), prepared using two methods in several stages. At the first stage, the CNT/PANI composite was synthesized by *in situ* polymerization of aniline in the presence of MWCNTs. Next, microfibers based on MWCNT / PANI composite were electrospun, where 1D MWCNT/PANI particles were aligned parallel to each other. This technique allowed to obtain a material with a highly ordered structure. The conductivity and Seebeck coefficient in this composite were 17.1 S/cm and 10 $\mu\text{V}/\text{K}$, respectively, and the power factor was 0.171 $\mu\text{W}/(\text{m}\cdot\text{K}^2)$. A porous PANI layer was formed in [8] on the surface of the MWCNT, allowing to increase the Seebeck coefficient to 79.8 $\mu\text{V}/\text{K}$ with a virtually unchanged conductivity of 14.1 S/cm, providing a significant increase in the power factor to about 9 $\mu\text{W}/(\text{m}\cdot\text{K}^2)$. The authors believe that the presence of pores 10–20 nm in size (comparable to the mean free path of phonons in PANI) leads to a significant increase in phonon scattering and, as a result, to a decrease in the thermal conductivity of the composite.

Composites based on single-walled carbon nanotubes (SWCNTs) and PANI exhibit considerably higher electrical conductivity and Seebeck coefficient [9–11] than MWCNT/ PANI composites. The power factor of SWCNT/ PANI composites was, according to the estimates in [11], 175 $\mu\text{W}/(\text{m}\cdot\text{K}^2)$, which is the current record for P among organic thermoelectric composites and is comparable to the value of the power factor in inorganic thermoelectric materials. It is hypothesized that the reason for the increase in the specific conductivity and the Seebeck coefficient in these composites is the strong interaction between $(\pi-\pi)$ -conjugated bonds of carbon nanotubes and polyaniline molecules, serving to produce a more ordered structure of polymer chains along the nanotube.

The goal of this study is to monitor the variation in the thermoelectric parameters of the MWCNT/PANI composite in a wide temperature range, evaluating the effect of polyaniline doping on the thermoelectric characteristics of the experimental samples.

We expect that achieving this goal will allow to establish the optimal conditions for fabricating the composite with the highest value of the power factor.

Experimental samples and measurement procedure

We used multiwall carbon nanotubes obtained by catalytic pyrolysis of hydrocarbons, 17 ± 5 nm in diameter and 10 μm in length. MWSNTs were not aggregated; they entangled chaotically, forming a homogeneous material. The chemicals (aniline and ammonium persulfate from Fluka, USA) were used without prior purification. Fixed-pH solutions were prepared using 0.1 n of standard fixing agents and distilled water.

PANI was applied to the surface of MWCNTs by heterophase synthesis (*in-situ* polymerization), via oxidative polymerization of aniline in the presence of MWCNTs dispersed in the reaction medium. The reaction was carried out in a water/alcohol solution mixed with ammonium persulfate at 0 °C and stirred vigorously. After the synthesis was finished, the product was decanted into a filter and washed first with an acidified aqueous solution, and then with acetone. It was then dried under normal conditions until the weight of the product was stabilized. The PANI content in the composition of the material was found from the increase in the weight of the composite, which correlated well with the 100% yield of the polymer during synthesis. The reference material used was PANI synthesized under the synthetic conditions but in the absence of MWCNTs.

PANI was synthesized in an oxidized electrically conductive form. The decrease in conductivity was controlled by treating the MWCNT / PANI composite with buffer solutions with specific pH. The material was then filtered from the buffer solution and dried to constant weight.

The morphology of the materials was studied via of NANOSEM 450, a scanning electron microscope (SEM) from by Fei (USA), at an accelerating voltage of 5 kV and a pressure of 90 Pa.

The MWCNT/ PANI composites were compressed into pellets 16 mm in diameter and 1–3 mm in thickness under 392 MPa for thermoelectric measurements.

Conductivity and Seebeck coefficient were measured with the NETZSCH SBA 458 Nemesis setup (Netzsch, Germany). A four-point method was used to measure the specific conductivity of the sample. The temperature gradient for measuring the Seebeck coefficient was generated by means of two microheaters located at the edges of the sample. The quantities α and σ were measured automatically, alternately for each temperature point (with a constant step). The measurement accuracy was 7% for the Seebeck coefficient, and 6–7% for specific conductivity.

Experimental results and discussion

A typical morphology of PANI produced by template-free polymerization (particularly carbon nanotubes), serving as a reference material, is shown in Fig. 1, *a*. The polymer has a hierarchical structure. The first hierarchical level formed by self-assembly of insoluble aniline oligomers consisted of spherical particles about 10 nm in diameter. They assemble into quasi-spherical agglomerates of submicron sizes during synthesis (the agglomerates are clearly visible in the figure). These agglomerates then stick together to form shapeless micron-sized particles, which then precipitate into powdered polymer material [13].

If the template has a large specific surface area, which is the case with MWCNTs, rather than self-assemble into nanospheres, the oligomers are adsorbed onto the surface of the MWCNT, followed by heterophase growth of the polymer shell covering the surface of carbon tubes.

Fig. 1, *b, c* shows images of the original carbon nanotubes (MWCNT) and the MWCNT/ PANI composite. The initial carbon material is a homogeneous porous mat of chaotically entangled MWCNTs (Fig. 1, *b*). The overall structure of the material did not change when PANI was deposited, but the diameter of the composite tubes nearly doubled (Fig. 1, *c*), so the thickness of the PANI shell can be estimated at about 10 nm. Evidently, the structure is homogeneous and extended. The entire material has a nanocable-like morphology. The nanotubes are randomly oriented relative to each other.

Fig. 2 shows the temperature dependences for conductivity σ , Seebeck coefficient α and power factor of the MWCNT/ PANI composites treated with buffer solutions with different pH values. As evident from Fig. 2, *a*, the maximum conductivity is observed in the composite treated with the most acidic buffer solution with the pH value = 1.0. An increase in the hydrogen index of the buffer solution leads to an uneven decrease in the conductivity of the composite over the entire temperature range considered. For example, an increase in the pH of the buffer from 1.0 to 2.0 at a temperature of 296 K provides a decrease in specific conductivity from 32 to 22 S/cm, while an increase in the pH from 3.5 to 7 is accompanied with a decrease in the conductivity from 18.5 to 16.5 S/cm. It is known for buffer pH values of 1.5–2.0 that PANI films show a sharp transition from the basic emeraldine form (when it is a dielectric) to doped acidic emeraldine form, when its conductivity increases sharply [14]. However, it is noteworthy in this case that the treating the MWCNT / PANI composite in acidic (pH = 1.0) and neutral (pH = 7) buffers only produces a twofold change in its conductivity, while treating the powder or a sufficiently thick PANI film in solutions with the same acidity reduces its conductivity by several orders of magnitude [14, 15].

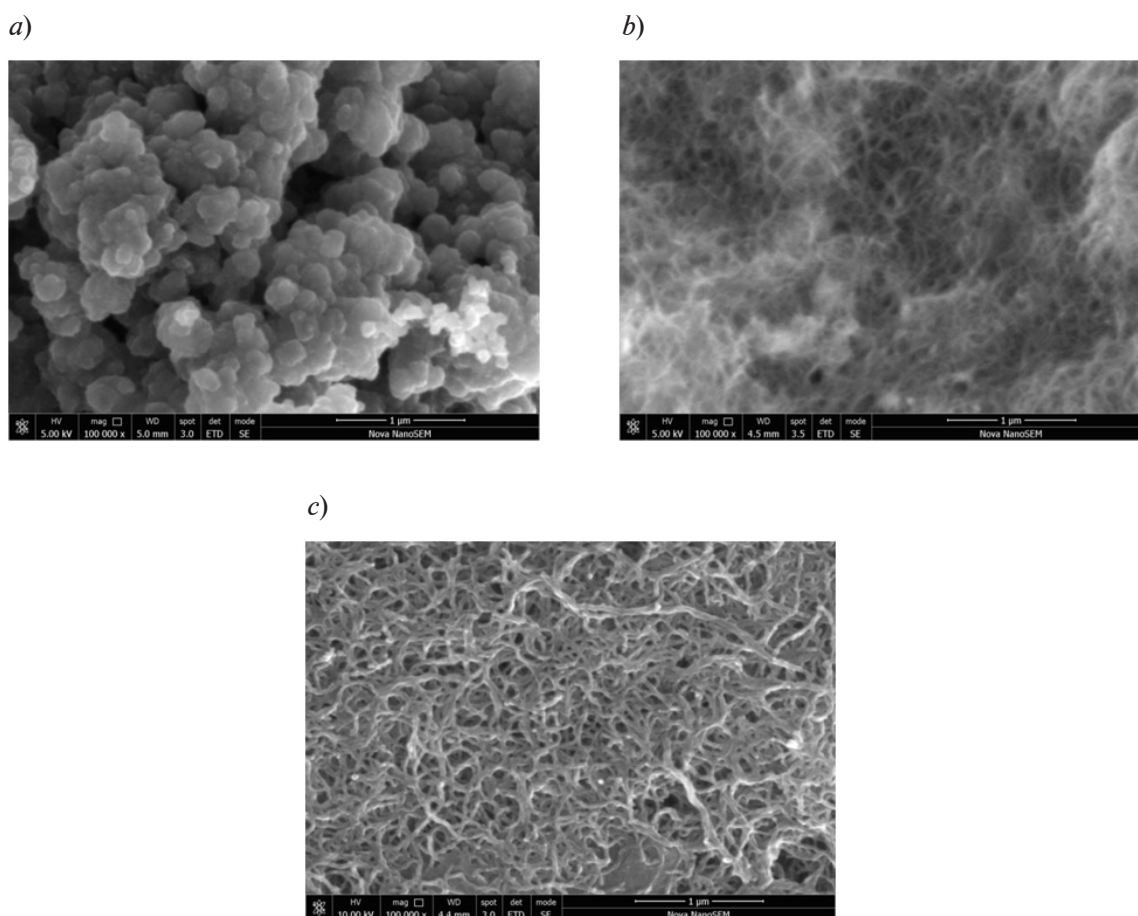


Fig. 1. SEM images of polyaniline (reference material) obtained in the absence of MWCNTs (*a*); initial carbon nanotubes (*b*) and the same nanotubes after PANI deposition (*c*). All scale bars are equal to 1 μm

Studies of electrical conductivity of PANI established that the contact between aggregates at the last hierarchical level is limited to transport of charge carriers [16]. Such aggregates are shapeless unconsolidated particles of micron sizes, formed primarily during PANI sedimentation after the synthesis is completed. The highest electrical conductivity is observed in nanospheres, which are particles of the first hierarchical level where polymer chains are densely packed during synthesis. Dedoping the micron particles critically reduces the electrical conductivity of the material. On the other hand, dedoping of densely packed spheres only occurs at the edges of the particles and does not lead to a significant decrease in their conductivity. Apparently, the shell of the MWCNT/PANI composite is a densely packed layer of macromolecules, similar to particles of the first hierarchical level. It has increased electrical conductivity and is weakly dedoped, so the conductivity of MWCNT / PANI decreases slightly with an increase in pH.

The temperature dependences of conductivity exhibit different behaviors in composite samples treated with buffer solutions with different pH values. When the composite is treated with a buffer with pH = 7, the temperature only slightly affects the specific conductivity, which increases approximately linearly from 16.5 to 17.5 S/cm with an increase in temperature from 295 to 413 K. The linear nature of the $\sigma(T)$ dependence is preserved at pH = 6.0, but the slope of the line increases markedly, i.e., heating of the sample produces a more significant increase in its conductivity. Starting from pH = 3.5, the $\sigma(T)$ dependence ceases to be linear, a peak starts to form on it (at $T \approx 370$ K), becoming pronounced for samples with high conductivity.

Repeating the heating cycle for a sample of highly conductive MWCNT / PANI composite to 413 K with subsequent cooling, we found that the $\sigma(T)$ dependences measured during heating and cooling do not coincide. Fig. 3 shows a graph of the $\sigma(T)$ dependence for the MWCNT/PANI

composite treated with a buffer solution with $\text{pH} = 1.0$, during heating and subsequent cooling. Evidently, the $\sigma(T)$ curve characterizing cooling passes below the curve corresponding to heating. The difference in σ values is about 2 S/cm. We can hypothesize that water and dopant losses in the samples may be the reason why the conductivity of the composite decreases upon heating above 370 K. Such a process may occur in PANI exposed at elevated temperatures, as described in [17].

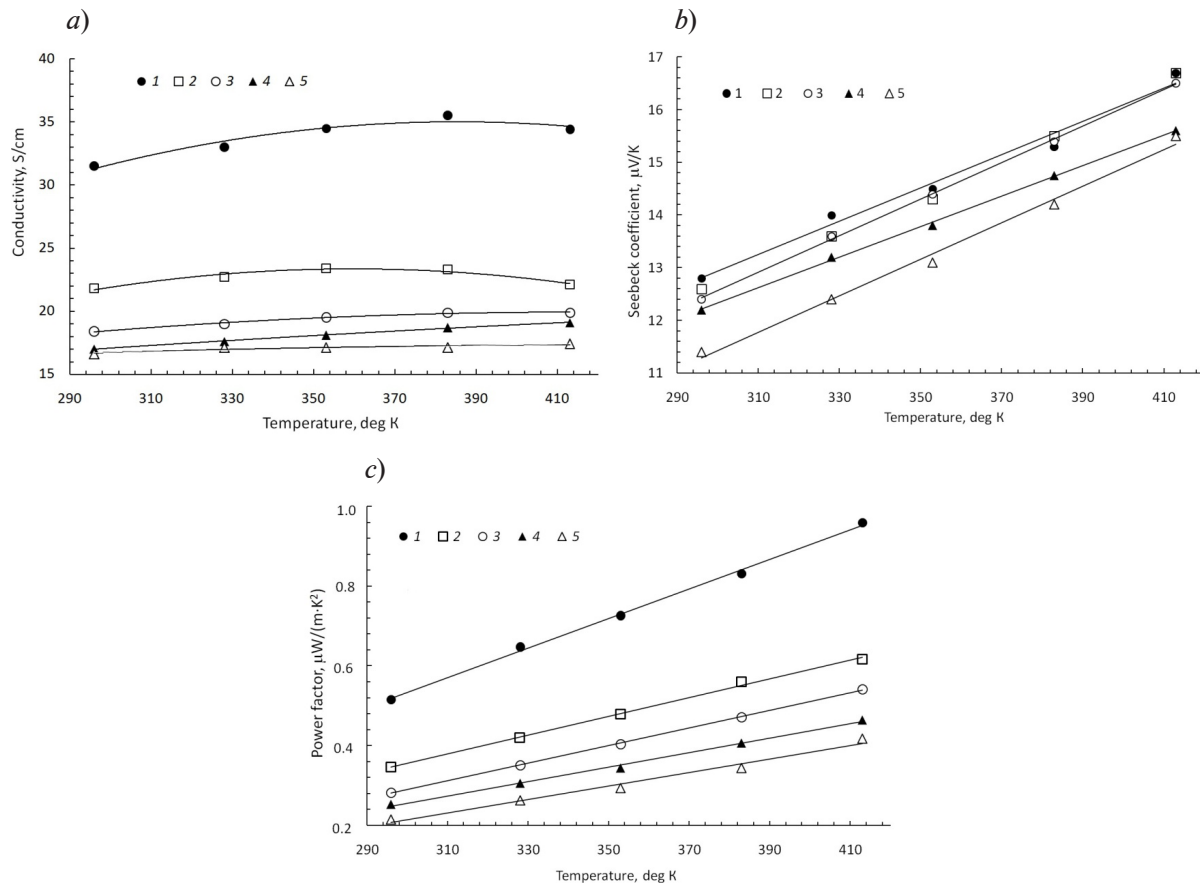


Fig. 2. Temperature dependences of conductivity (a), Seebeck coefficient (b) and power factor (c) for samples of MWCNT/PANI composite after treatment with solutions with different pH values: 1.0 (1), 2.0 (2), 3.5 (3); 6.0 (4) and 7.0 (5)

Fig. 2, b shows the temperature dependence for the Seebeck coefficient of the given samples treated with buffer solutions with different pH. The value of α exhibits linear growth over the entire temperature range considered for all pH values. The increase in the Seebeck coefficient relative to the initial value at $T = 413$ K amounted to approximately 30%. As seen from the graph, the highest Seebeck coefficient in the entire temperature range is observed in the sample with the highest conductivity, treated with a buffer solution with $\text{pH} = 1.0$. As the pH increases, the value of α decreases.

Based on the measurement results obtained for $\sigma(T)$ and $\alpha(T)$, we calculated the temperature dependences of the power factor for all samples (see Fig. 2, c). Apparently, regardless of the doping degree, an increase in temperature leads to an increase in P by a law close to linear. The highest value of the power factor is characteristic for the composite material treated with a buffer solution with $\text{pH} = 1.0$. $P \approx 0.5 \mu\text{W}/(\text{m}\cdot\text{K}^2)$ for this sample at room temperature, while this coefficient increases to about $1 \mu\text{W}/(\text{m}\cdot\text{K}^2)$ at 413 K. However, its thermoelectric characteristics gradually deteriorate over time at such a high temperature, due to loss of dopant from the sample. Therefore, despite a significant increase in the power factor, it seems impractical to use this composite at temperatures above 370 K.

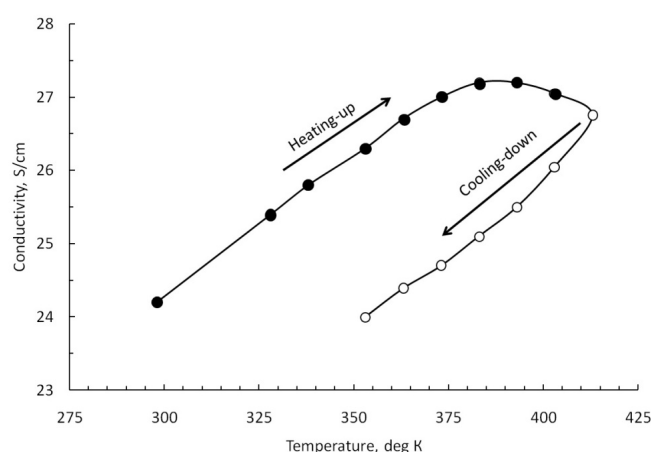


Fig. 3. Variation in specific conductivity of MWCNT/PANI composite sample treated with a buffer solution with $\text{pH} = 1.0$ during heating and subsequent cooling

Conclusion

Thus, considering the effect of temperature on the MWCNT/PANI composite material at varying doping concentrations of PANI, we found that the composite with the highest conductivity exhibited the greatest power factor equal to $0.5 \mu\text{W}/(\text{m} \cdot \text{K}^2)$ at room temperature. Comparing the obtained results with the data available in the literature, we can conclude that the power factor of this sample, characterized by chaotic arrangement of nanotubes, substantially exceeds the value of P for the MWSNT/PANI composite, obtained in [10] via a far more complex technology, that is, electrospinning, which achieves ordering of MWSNTs relative to each other.

Even though the magnitude of P in the samples considered grows with increasing temperature ($P \approx 0.8 \text{ W}/(\text{m} \cdot \text{K}^2)$ for $T = 370 \text{ K}$), it seems ill-advised to use this composite at 370 K, since it can be assumed that the dopant gradually withdraws from the composite at high temperatures, and its conductivity decreases.

As the following step, aimed at improving the thermoelectric characteristics of the MWSNT/PANI composite, we plan to develop the technology for producing samples with increased porosity of PANI coating on MWSNT surface.

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THE AUTHORS

TRETYAKOV Artem A.

Peter the Great St. Petersburg Polytechnic University
29 Politechnicheskaya St., St. Petersburg, 195251, Russia
tretartem@gmail.com
ORCID: 0000-0003-1009-8016

KAPRALOVA Viktoria M.

Peter the Great St. Petersburg Polytechnic University
29 Politechnicheskaya St., St. Petersburg, 195251, Russia
kapralova2006@yandex.ru
ORCID: 0000-0001-9050-4453

LOBODA Vera V.

Peter the Great St. Petersburg Polytechnic University
29 Politechnicheskaya St., St. Petersburg, 195251, Russia
vera_loboda@mail.ru
ORCID: 0000-0003-3103-7060

SAPURINA Irina Yu.

Institute of Macromolecular Compounds RAS
31 Bolshoy Ave. V. Isl., St. Petersburg, 199004, Russia
sapurina@mail.ru
ORCID: 0000-0001-7579-7577

SUDAR Nikolay T.

Peter the Great St. Petersburg Polytechnic University
29 Politechnicheskaya St., St. Petersburg, 195251, Russia
sudar53@mail.ru
ORCID: 0000-0001-7380-7727

**СВЕДЕНИЯ ОБ АВТОРАХ**

ТРЕТЬЯКОВ Артем Александрович – аспирант Высшей школы электроники и микросистемной техники Санкт-Петербургского политехнического университета Петра Великого.

195251, Россия, г. Санкт-Петербург, Политехническая ул., 29

tretartem@gmail.com

ORCID: 0000-0003-1009-8016

КАПРАЛОВА Виктория Маратовна – кандидат физико-математических наук, доцент Высшей школы электроники и микросистемной техники Санкт-Петербургского политехнического университета Петра Великого.

195251, Россия, г. Санкт-Петербург, Политехническая ул., 29

kapralova2006@yandex.ru

ORCID: 0000-0001-9050-4453

ЛОБОДА Вера Владимировна – кандидат физико-математических наук, директор Высшей школы электроники и микросистемной техники Санкт-Петербургского политехнического университета Петра Великого.

195251, Россия, г. Санкт-Петербург, Политехническая ул., 29

vera_loboda@mail.ru

ORCID: 0000-0003-3103-7060

САПУРИНА Ирина Юрьевна – доктор химических наук, ведущий научный сотрудник Института высокомолекулярных соединений РАН.

199004, г. Санкт-Петербург, Большой пр. В. О., 31

sapurina@mail.ru

ORCID: 0000-0001-7579-7577

СУДАРЬ Николай Тобисович – доктор физико-математических наук, профессор Высшей школы электроники и микросистемной техники Санкт-Петербургского политехнического университета Петра Великого.

195251, Россия, г. Санкт-Петербург, Политехническая ул., 29

sudar53@mail.ru

ORCID: 0000-0001-7380-7727.

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