

*Підвищення ефективності протиоблідних засобів авіакосмічної техніки базується на створенні високоефективних нагрівачів. Найбільш затребуваними є електронагрівачі з ефектом саморегулювання температури за рахунок позитивного або негативного температурного коефіцієнта опору. Для розроблення нагрівачів із заданими властивостями використовуються різні типи матриць на основі цементу, скляної фритти, асфальтової мастики або полімерів. Найбільш ефективними є вуглецеві наноструктури. Залежно від технології отримання вуглецевих наноструктур, а також особливостей композитів, у які вносяться провідні структури, визначаються основні властивості електронагрівачів. Для дослідження ефективності електронагрівачів була використана методика на основі безконтактного методу вимірювання температурного поля. Синтез ВНТ відбувся на Ni/MgO каталітичній системі, отриманий методом термічного розкладання. Морфологію ВНТ вивчено за допомогою польового емісійного електронного мікроскопа Hitachi H-800. Для електронагрівача базова питома потужність складає  $800 \pm \pm 10$  % Вт/м<sup>2</sup> при температурі навколишнього середовища +10 °С. При зниженні температури навколишнього середовища до -40 °С питома нагрівальна потужність складає  $1600 \pm 20$  % Вт/м<sup>2</sup>. Динамічна зміна потужності при різних температурах свідчить про ефект саморегулювання. Із термограм визначено, що стабілізація тепловиділення відбувається при температурі 56 °С. Розроблені електронагрівачі можуть працювати при нарузі живлення до 200 В та мають раціональні електрофізичні та функціональні параметри, які дозволяють ефективно працювати в противооблідних засобах авіаційної техніки*

*Ключові слова: електронагрівач, вуглецеві нанотрубки, саморегулювання, теплообмін, парафін, самовстановний тепловий контакт*

## 1. Introduction

Electric heaters find use in various fields, including chemical and aerospace industry and household [1, 2]. Electric heaters can be used for local heating and for heating of large objects. Wide application of electric heaters is due to the ability for gradual control of heating power and rather

precise control over sustained temperature, simple installation, and good maintainability. The functionality of electric heaters is based on heat generation according to Joule-Lenz law [3]. The law states that the amount of heat produced by the electric heater is proportional to the square of electric current passing through it. There is not much difference between the operation on direct and alternating current,

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# ELECTRIC HEATERS BASED ON NANOMODIFIED PARAFFIN WITH SELF-INSTALLING HEAT CONTACT FOR ANTI-ICING SYSTEMS OF AEROSPACE CRAFTS

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with exception of some effects related to alternating current. Namely, the formation of electromagnetic fields in coiled heaters and related to the active inducted characteristics of electrical resistance.

Initial manufacturing technologies of electric heaters based on metal alloys such as nichrome, kanthal, and constantan were sufficient to sustain various demands for electric heating. High resistance alloy is used for manufacturing of heating elements that can be used for gaseous and air media. However, the reliability of such elements is rather low. This has led to the use of ceramic, composite and polymer materials. In turn, ceramic material could be with or without self-regulation. Acquisition of self-regulation was important for new types of heaters based on composites and polymers.

The self-regulation opened fundamentally new horizons for development and application of electric heating. Primarily it allowed for operation without automation and control mechanism. At the same time, the operation of self-regulating heaters is largely influenced by mechano-thermal removal of heat contact. In order to eliminate this effect, it is important to evaluate the possibility of using materials that would provide compensation and optimization of heat contact. This is especially important for electric heating systems used in aerospace crafts.

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## 2. Literature review and problem statement

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Improving the safety of aerospace crafts is related to the use of electro-thermal IPS (ice protection systems). Electro-thermal IPS are employed in cases when engines of aerospace crafts are sensitive to supplied air or there are difficulties in routing hot air pipes to protected surfaces. The power supply for electro-thermal IPS is craft's electric grid (115/208 V). Standard heating element for electro-thermal IPS is a wire with high specific resistance, foil and conductive films [4]. The paper [5] describes that creation of effective electro-thermal IPS requires optimization of heating surface.

However, standard technologies that are used for the creation of IPS based on electric heaters utilizing high resistance metallic conductors possess low energy efficiency as they cannot provide uniform heating of optimal surface [5]. This is because the passage of current through the conductor results in its heating and heat radiation into the environment, and if heat dissipation is not intensive the entire heating power is used for constant temperature increase. Many technological processes require strict temperature regime. This is particularly important for aerospace crafts where saving each watt of electricity matters. Various automation systems – sensors and regulators are used, in order to control and automatically increase or decrease power. Additional problems are related to the choice of control mechanism «P», «PI» and «PID». Overheating of heaters eventually results in their destruction.

It should be noted that materials capable of changing their volume during heating can also serve as an actuator, mechanically displacing ice [6].

Heating element for electro-thermal IPS is required to be reliable, repairable and capable of heating large areas. There are many technical solutions for electric heating. Metallic resistive heaters don't fulfill these requirements entirely. This is because of difficulties in the organization of thermal contact, reliability and uniform distribution of

temperature field. A new approach to solving the problems of transforming electrical energy into heat is based on principals of inductive heating [1]. Application of induction heating allowed solving the problem of heat contact as the electric current is induced directly into the heating surface. However, implementation of induction heating requires complex equipment. This method is also not suited for heating the majority of horizontal surfaces. Search for a simple and effective way for transforming electric energy into heat led to the development of electrically conductive composites. Various types of materials are used for the creation of conductive composites. The main focus is on materials that are technologically suited for manufacturing.

The paper [7] proposes a variant of an electric heater that is based on spinels of Mn, Co and Ni oxides (Mn 1.85; Co 0.8; Ni 0.35) O<sub>4</sub>, RuO<sub>2</sub>, with glass frit as a binder. The resulting material possesses a negative temperature coefficient. The major disadvantage of such heater is manufacturing complexity. Operating voltage is up to 46.7 V and heating temperature reaches 340 °C, which makes such heater unsuited for application in IPS.

Unlike ceramic heaters, which degrade under mechanical and temperature influence, cement-based heaters can be significantly more reliable. Let's conduct an analysis of modern approaches to the development of heaters based on cement matrixes.

The paper [2] studies the possibility of creating cement-based heaters. The conductivity of cement composites is achieved with the use of nickel particles. The authors of the paper [2] have studied cement composites with various types and content of nickel particles. Experimental results reveal that cement composites with nickel particles can be heated up to 50 °C over 30 s at a voltage of 20 V. With the input voltage of 15 V, cement composites with nickel particles (12 vol. %, 2.6–3.3 μm diameter) can melt 3mm of ice for 478 s at an ambient temperature of –16 °C.

The major disadvantage of such heaters is their brittleness and low operating voltage, which results in high current in power supply wires.

Another technology related to cement heaters is the use of carbon materials as conductor [8]. The use of carbon additives requires a large concentration of fine carbon powder, which impacts the durability of the cement composite. To solve the problem of reducing the concentration of the conductive phase, the paper [9] proposes the inclusion of carbon fiber into the cement matrix. Experiments conducted in the paper [9] resulted in conductive fiber-reinforced concrete. The results of the paper [9] revealed that the use of wet cement mixture provides better formability and good fiber dispersion. Additionally, the specific resistance is within 3–0.6 Ohm at the contents of fibrous carbon from 0.2 to 0.8 % vol.

Brittleness of the reviewed composites [2, 8, 9] leads to a review of more flexible and ductile matrixes. Such a matrix is asphalt mastic, composed of bitumen and mineral fillers. The paper [10] describes the use of carbon fiber and carbon power in order to make asphalt mastic. Target application of this heating composite is ice protection systems. The composite with carbon fiber and graphite powder showed the non-linear dependency of resistance on temperature [10]. However, the described composite possesses low specific energy parameters.

It is worth to note that for application in electric heaters, carbon fibers possess good conductivity and thermal stability. The authors of the paper [11] describe the heater with the

deliberately altered microstructure of carbon fiber through deposition of carbon atoms. In the microstructure of deposited carbon atoms, the initial layer is formed by small domains, an intermediate layer with average crystallinity and the final layer with large domains with a high degree of crystallinity. However, a linear decrease of conductivity in heating elements is due to the change in crystal orientation from linear to perpendicular relative to fibers. The output power of such composites ranges from 270 to 448 W when fed with 50 V. This technology can be used for various technical applications [11]. Manufacturing complexity limits its application for heating large surfaces.

Solution to the problems of creating heaters with the large heating surface can lie in the use of polymer matrixes. Heaters based on polymer composite materials with carbon nanotubes (CNT) are considered promising. This provides a high thermal conductivity of CNT while the low heat capacity of the polymer allows for fast heating [12]. One of the options for creating polymer matrixes for electric heaters is a modification of polydimethylsiloxane (PDMS). The electrical conductivity of CNT/PDMS with higher CNT contents was higher by two orders than those with lower CNT content [12].

The author of the paper [13] describes an electric heater based on a composite of high-pressure polyethylene with graphite nanofibers and carbon soot. The disadvantage of this heater is the upper voltage limit of 100 V.

The paper [14] describes the use of polypropylene with a hybrid filler of graphene oxide and multilayer CNT (binary filler) and multilayer carbon nanotubes. The use of the binary filler can result in non-uniform electrophysical properties over the volume of the electric heater.

The use of polypropylene [15] allows for the creation of a heater with operating voltages above 100 V. The use of the binary conductive filler at a concentration of 1.5–2 wt. % can result in a non-uniform distribution in the dielectric matrix.

The authors of the paper [16] have studied flexible heating elements based on silicone. An alloy with a platinum catalyst and titanium oxide, coated with tin oxide and antimony and carbon soot were used as fillers. The resulting heater had constant volume making it unusable as an actuator, which could've provided mechanical removal of ice.

The paper [17] describes the development of CNT-based heaters with a positive temperature coefficient (PTC). PTC composite [17] was prepared from high-density polyethylene and CNT as a conductive phase. The paper [18] described the use of graphene nanoplates as an electrically conductive additive to the polymer composite. The heater with graphene nanoplates demonstrates the heating rate of 25 °C/min at 3–5 V. The material was also flexible, owing to the content of the conductive phase 3–5 wt. %. However, the major disadvantage of described heaters [17, 18] is low operating voltage, as IPS requires heaters with a working voltage of 110 to 220 V.

The influence of CNT on PTC of the heater with the polymer matrix is studied in the paper [19]. The authors have studied the relation between the thermal expansion of polyvinylidene fluoride (PVDF) and PTC-effects in PVDF/CNT and PVDF/carbon soot. PTC effects were observed at temperatures below the melting point of the polymer. A more detailed study of this effect is described in the paper [20]. PTC changes in composites with Ni content of 40 and 50 vol. % occurs at temperatures close to the melting point

of PVDF [20], while composites with Ni content of 20 and 30 vol. % PTC effect manifested at a temperature below the melting point of the PVDF matrix. The authors have discovered that PTC effect occurs even without melting of the polymer. A small increase of specific volume at temperatures below the melting point of the polymer results in a gap between the conductive filler which affects electric resistance.

The key problem with heating of a complex object in a rapidly changing temperature regime is drifting of heat contact or heat exchange surface. Under conditions of drifting heat content, the heaters capable of sustaining constant temperature on their surface are most susceptible to loss of effectiveness. This negative effect can be avoided with the use of heaters that can undergo volume change to a degree that would compensate for thermal drift of heat exchange surfaces. Development of such materials is most effective with the use of carbon nanostructures that can change their volume during heating.

Thus, there are many technologies for the creation of heating composites. They differ in conductive components, binding matrixes, and manufacturing processes. This allows for the creation of many electrophysical parameters and operation regimes for composite electric heaters. At the same time, there are strict requirements for heaters of IPS in terms of weight, size, feed voltage, and additional functionalities. Among new functionalities are self-installing thermal contact and actuator effect. From the literature review [1–20], it can be concluded that in the field of composite electric heaters, the problems of forming effective heat contacts between the heating surface and heated object are not well studied and almost ignored.

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### 3. The aim and objectives of the study

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The aim of the work is to evaluate the possibility of improving the efficiency of self-regulating heaters, by using the composite matrix based on paraffin modified with CNT.

To achieve the set aim, the following objectives were formulated:

- to prepare samples of heaters based on paraffin modified with carbon nanotubes;
- to study the heating power of the heater based on nanomodified paraffin when fed with a constant voltage;
- to conduct a comparative analysis of the influence of heat contact on the operation of heaters with matrixes based on polyethylene and paraffin.

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### 4. Materials and methods for preparation of heaters and study of their characteristics

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#### 4.1. Heater preparation

Paraffin P-2 (Lukoil, Russia) was used for heater construction. CNT were used for paraffin modification. Nickel foil with 10 µm thickness was used for voltage supply. The heater constructed from nanomodified polyethylene was used for comparison.

#### 4.2. Synthesis of carbon nanotubes

CNT were synthesized using the thermal decomposition method with Ni/MgO catalyst. Initial components



( $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , analytic grade glycine) were dissolved in water at 50–60 °C.

CNT morphology was studied using data recorded using the field emission scanning microscope Hitachi H-800 (Hitachi, Japan) in a vacuum under  $6 \cdot 10^{-5}$  Pa and voltage of 20 kV.

#### 4. 3. Paraffin nanomodification

Paraffin at 50 °C was poured into a silicone vessel. Then, CNT (2.75 % wt.) was added through metallic sieves (cell size 40  $\mu\text{m}$ ). The mixture was irradiated with 22 kHz ultrasound and mechanically stirred (50 rpm) at a constant temperature of 50 °C. The obtained suspension was poured on flat glass and dried under vacuum for 3 h.

#### 4. 4. Assembly of the heater based on nanomodified paraffin

The film of nanomodified paraffin (functional material of the heater) with a thickness 3 mm was placed between two nickel plates 100  $\mu\text{m}$  (current collectors). Thermal insulation based on microspheres (Isolate, Russia) was used as heat insulation for the opposite side of the heater. The assembled heater was then sealed in a dielectric frame made of fluoropolymer film.

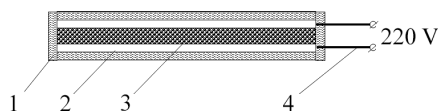


Fig. 1. Schematic of a self-regulating heater:  
1 – dielectric frame; 2 – current collectors;  
3 – functional material; 4 – current feed conductors

Heat exchange with the environment or heated surfaces occurs through the dielectric frame. Heat flow occurs on the side that is not thermally insulated.

#### 4. 5. Study of self-installing heat contact

Fig. 2 shows a device that was used to study the self-installing heat contact. The device includes a Peltier element. On one side of the Peltier element – heater, and on another – passive aluminum radiator.

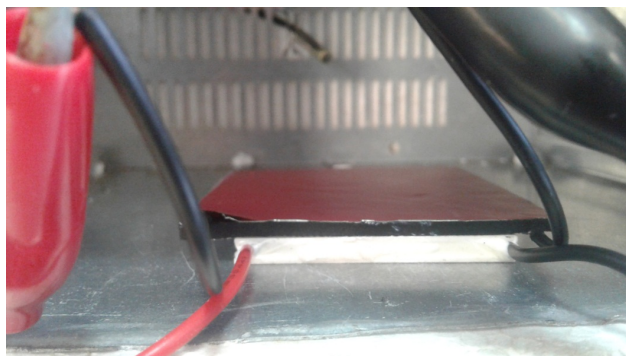


Fig. 2. Plateau with Peltier element – heat-exchanger with axial fan is installed at the lower part of Peltier element

For studying the self-installing heat contact, the lower Peltier element is supplied with 10 V, which results in a temperature decrease to 10 °C over 10 min. The heater is turned on (it is fed with constant 200 V). The weight of 50 grams is then placed onto the heater. Heater's power

consumption is measured using the multimeter UT-71E (Uni-T, China).

The thermal field on the heater surface and in the contact zone was studied using the thermal imager Testo-875 (Testo, Germany). The error for temperature regime measurements is within 0.2 °C. The error for power consumption is within 0.2 W. All experiments were conducted under identical conditions and were repeated 10 times. The data presented in the graphs is an average of 10 tests. The error is additive over the whole range of measurements.

### 5. Results of studying the operation of the heater with self-installing contact

All synthesized samples are mainly composed of fiber-like formations (nanotubes) Fig. 3.

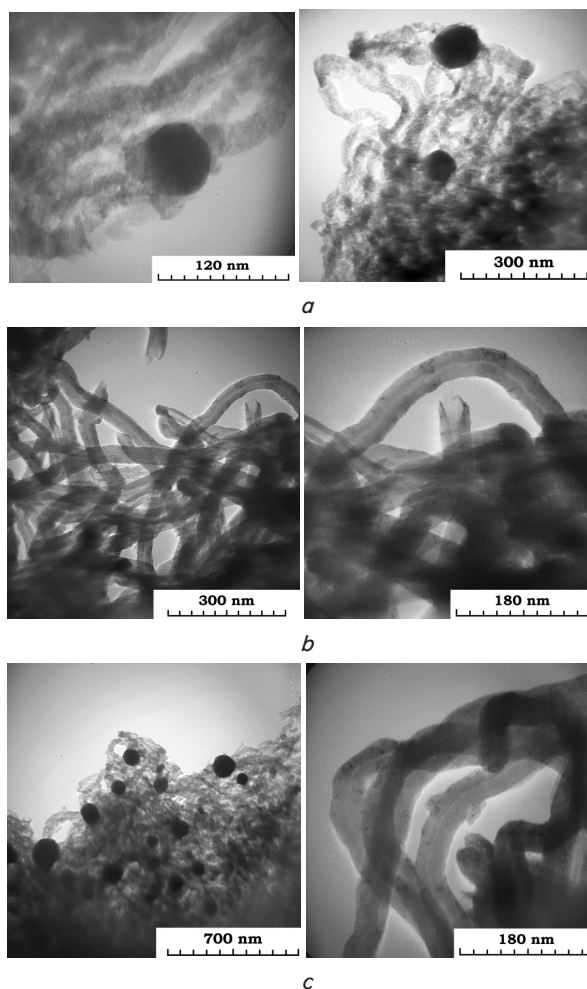


Fig. 3. TEM images of the samples on the catalyst:  
*a* –  $\text{Ni}/_{0.3}\text{MgO}$ ; *b* –  $\text{Ni}/_{0.5}\text{MgO}$ ; *c* –  $\text{Ni}/_{0.16}\text{MgO}$

#### 5. 1. Results of studying the power of the heater based on nanomodified paraffin when fed with a constant voltage

The power of the self-regulating heater at different ambient temperatures (Fig. 4). It should be noted that higher concentrations of CNT in the polymer matrix shifts the power line to the right and up.

Dynamics of the increasing power of the self-regulating heater with increasing concentration of CNT at minus 40 °C (Fig. 5).

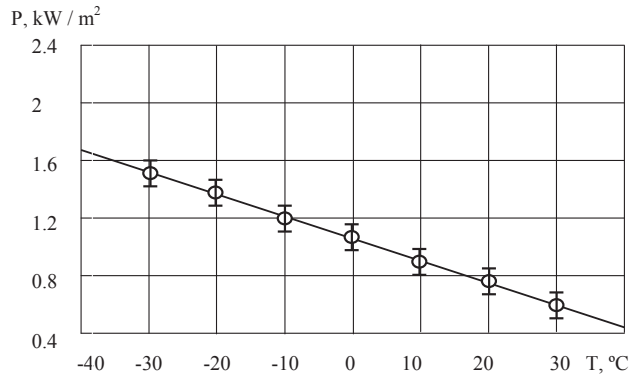


Fig. 4. Dependence of the power of the self-regulating heater on ambient temperature

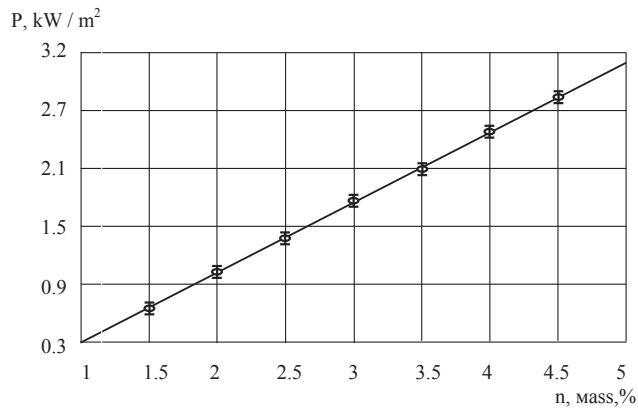


Fig. 5. Dependence of heater power on CNT concentration (1; 1,5; 2; 2,5; 3; 3,5; 4; 4,5; 5 % wt.)

The approximated dependence of power on CNT concentration is expressed by the equation:  $P(n)=0.72n-0.44$ . Determination coefficient is equal to 0.99, and average approximation error is 3.47 %.

Fig. 6 shows a thermal image of the electric heater at an operating voltage of 200 V.

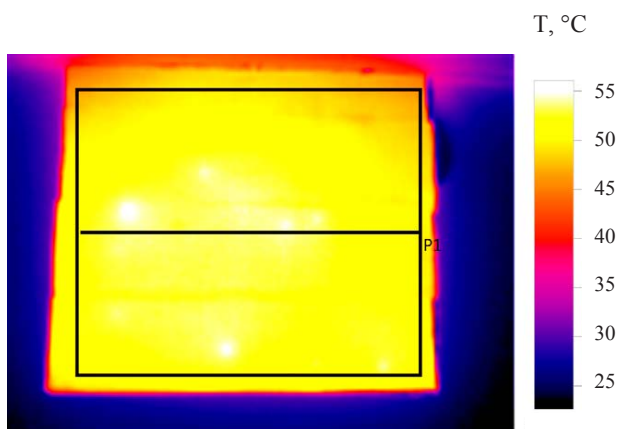


Fig. 6. Thermal image of the surface of the self-regulating heater

### 5. 2. Results of studying the influence of thermal contact on the operation of heaters based on polyethylene and paraffin

Fig. 7, a, b shows thermal images of thermal contact between the heater’s surface and the heat exchanger.

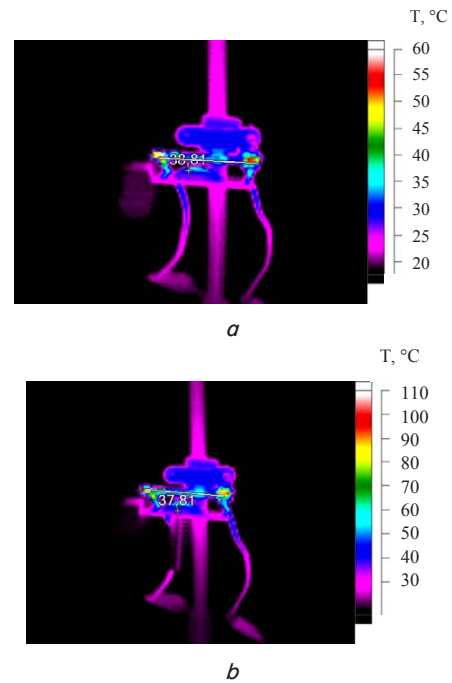


Fig. 7. Distribution of temperature field in the heater-heat exchanger system: a – heater based on nanomodified polyethylene; b – heater based on nanomodified paraffin

Fig. 8 shows a thermal profile in the heater – heat exchanger contact zone:

- a) heater based on nanomodified polyethylene;
- b) heater based on nanomodified paraffin, after 30 s of being turned on. The thermal profile is derived from data shown in Fig. 7.

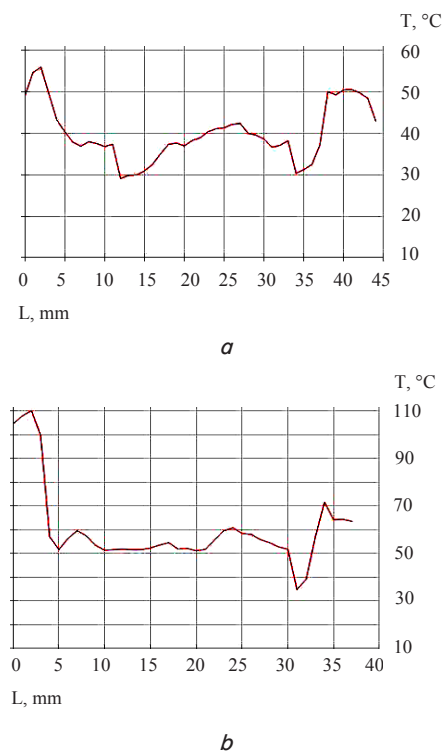


Fig. 8. Temperature profile in the heater – heat exchanger contact zone: a – heater based on nanomodified polyethylene; b – heater based on nanomodified paraffin

Comparison of the temperature profile shown in Fig. 8, *a*, *b*; reveals that for nanomodified paraffin there is a stabilization of the temperature regime in the cross-section between the heater and the heat exchanger. This can be explained by improved contact in the heater-heat exchanger system.

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## 6. Discussion of the results of electric heaters with self-installing contact

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Materials synthesized on Ni<sub>0.3</sub>MgO and Ni<sub>0.16</sub>MgO catalysts exhibit the most ordered and defined structure. The diameter of fibrous formations synthesized on Ni<sub>0.16</sub>MgO and Ni<sub>0.3</sub>MgO catalysts is ~30÷60 nm. The sample prepared on Ni<sub>0.5</sub>MgO catalyst contains not only carbon nanotubes but a significant amount of unreacted catalyst (Fig. 4). The heater based on nanomodified paraffin exhibits self-regulating properties as its output power changes with ambient temperature (Fig. 5). The basic specific output power is 800±10 % W/m<sup>2</sup> at an ambient temperature of +10 °C. When the ambient temperature is lowered to -40 °C, the specific heating power increases to 1600±20 % W/m<sup>2</sup>. The thermal image (Fig. 6) revealed that stabilization of heat output occurs at 56 °C and lowers the load in the craft's power grid. Mass to power ratio is controlled on the manufacturing stage of material. For the prepared electric heater, when self-regulation manifests, specific output power ranges from 300 W/ m<sup>2</sup> to 3 kW/m<sup>2</sup> at -40 °C (Fig. 5). Analysis of Fig. 7 and 8 revealed that after 30 seconds there is a phase transition in nanomodified paraffin with volume increase by 2 %. This results in a more secure contract with the heat exchanger. In this case, the change of the temperature profile is as shown in Fig. 7, *b* and Fig. 8, *b*. From the comparison of temperature regimes shown in Fig. 7, *a*, *b* and 8, *a*, *b*, it follows that there is an improvement of heat flow in the cross-section between the heater and the heat-exchanger for the variant with self-installing contact (*b*). This is the result of phase transition that occurs in nanomodified paraffin [21] and results in mechanical influence on the heated surface which allows for ice displacement. Temperature increases and stabilizes, which results in a stable heat flow. And the advantage of conducted experiments is the approach for studying the mechanism of the self-installing method with the use of non-contact measurements of the temperature field. For a more comprehensive study of heat mechanisms, through self-installing heat contact, it is necessary to conduct studies with a different area of heat transfer and heat output. Actuator effect – mechanical influence on the heating surface, also requires additional study.

The results obtained allow recommending electric heaters with self-installing heat contact for high-precision thermal stabilization and heating of elements with a large heat

transfer area. A possible application is the aerospace field (ice protection systems), where reliability and efficiency are of great importance along with low weight and size.

The research revealed that there is a potential related to the problems of self-installing contacts.

The present research is related to problems of electric heaters for ice protection systems. At the same time, the developed heaters can also be used for heating the plane's interior and telemetry sensors used in aviation. The paper does not study the problems related to the study of the influence of surface area on heat radiation regimes. Studies that would allow determining the influence of pressure on the heater's surface with changing the aircraft altitude are not present.

The main disadvantage of the study is that the mechanism of heat transfer through self-installing heat contact has not been studied in detail. This is important as the regime of electric heating is affected by heat transfer area and output power of the heater. Further development of this work can be studies regarding decreasing the size and weight of electric heaters and study of pressure effect on the heater's surface with changing the aircraft altitude. The main issue for this direction is possible issues in preserving the mechanical durability of electric heaters.

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## 7. Conclusions

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1. By employing nanomodification of paraffin with carbon nanotubes, a functional material which radiates heat upon passage of electric current was prepared. Upon installation of electrodes onto functional material and dielectric insulation, a heater is formed. The heater based on nanomodified paraffin showed self-regulating properties owing to the phase transition. The value of the specific surface area to power ranges from 300 w/m<sup>2</sup> to 3 kW/m<sup>2</sup>.

2. It was discovered that for the electric heater based on nanomodified paraffin under directed current, specific power is 800±10 % W/m<sup>2</sup> at an ambient temperature of +10 °C. When the temperature is decreased to -40 °C, specific heating power increases to 1,600±20 % W/m<sup>2</sup>.

3. It was discovered, that the effect of self-installing heat contact is manifested when the matrix capable of phase transition is used. Improvement of heat contact occurs within 20 seconds due to thermal expansion at a phase transition in nanomodified paraffin. This allows for improved heat transfer and efficiency of heaters used for large areas.

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