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TRANSPARENT HEATERS BASED ON THE COPPER MICROMESH PASSIVATED BY GRAF(PH)ENE OXIDE

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The paper presents the results of creating and researching the transparent heaters with enhanced performance characteristics. The heaters are based on a composite coating of a new type by contrast with classical solutions based on transparent conductive oxide films. Such a composite coating is copper micromesh obtained using a self-organized template, stabilized by a thin film of graphene oxide (GO). The micromesh coating is formed by magnetron sputtering of copper onto a template obtained as a result of self-organized cracking of a silica film. Then, a graphene oxide film protecting the micromesh coating from thermal and chemical degradation is applied to the micromesh coating by the spray-method. A composite coating with the surface resistance of 8.9 Ohm/sq is obtained with the transparency of 82.8 % at the wavelength of 550 nm. High uniformity of heating and stability of the composite coating are shown when operating under heating up to 97.2 °C for a long time (24 hours). The composite coating of the GO / Cu micromesh on a glass substrate 2 mm thick is characterized by the thermal resistance value of 134.2 °C·cm^2·W^{-1}, while the ITO literature sample on an equivalent substrate is characterized by the thermal resistance of 94.04 °C·cm^2·W^{-1}, which indicates higher heating efficiency at the same specific power dissipation. This fact opens up prospects for its use as an anti-icing coating in aerospace industry.

Keywords: transparent conductive coating, self-organized template, copper micromesh, transparent IR heater, graphene oxide.

ПРОЗРАЧНЫЕ НАГРЕВАТЕЛЬНЫЕ ЭЛЕМЕНТЫ НА ОСНОВЕ МЕДНОЙ МИКРОСЕТКИ, ПАССИВИРОВАННОЙ ОКСИДОМ ГРАФЕНА

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Представлены результаты по формированию и исследованию прозрачных нагревателей с повышенными эксплуатационными характеристиками относительно классических решений на основе оксидных плёнок. Композиционное покрытие представляет собой медную микросетку, полученную при помощи самоорганизованного шаблона, стабилизированную тонкой пленкой оксида графена (ОГ). Микросетчатое покрытие формируется посредством напыления меди на шаблон, полученный в результате самоорганизованного растрескивания пленки кремнезема. Затем на микросетчатое покрытие spray-методом наносилась пленка оксида графена, защищающая микросетчатое покрытие от термической и химической деградации. Получено композиционное покрытие с поверхностным сопротивлением 8,9 Ом/кв при прозрачности 82,8 % на длине волны 550 нм. Показана высокая однородность нагрева и стабильность композиционного покрытия при функционировании в условиях нагрева до 97,2 °С в течение длительного времени (24 часа). Композиционное покрытие ОГ / Cu-микросетка на стеклянной подложке, толщиной 2 мм, характеризуется значением теплового сопро-

тивления 134,2 °C·см^2·Вт$^{-1}$, в то время как литературный образец ITO на эквивалентной подложке характеризуется тепловым сопротивлением 94,04 °C·см^2·Вт$^{-1}$, что говорит о более высокой эффективности нагрева при одинаковой удельной рассеиваемой мощности, что открывает перспективы применения в качестве антиобледенительных покрытий в аэрокосмической отрасли.

Ключевые слова: прозрачное проводящее покрытие, самоорганизованный шаблон, медная микросетка, прозрачный нагреватель, оксид графена.

Introduction. Transparent heaters are used as anti-icing coatings applied in civil and military aviation, northern shipping, as well as in thermochromic windows. Taking into account the severe climatic conditions typical for our region, the development of cost-effective transparent heating elements is an important task for materials science.

It is also worth noting that in addition to their calorific value, transparent conductive coatings are used as transparent screens of radio emission, as well as transparent electrodes in solar cells and LEDs, which are integral parts of orbital stations and satellites.

Currently, transparent heaters based on indium oxide doped with tin (ITO) [1; 2] or tin oxide doped with fluorine (FTO) [3] are the most widely used ones, but they poorly meet the requirements put forward to the heating elements. Oxide transparent conductive coatings have low thermal conductivity, less than 10 W/m·K [4], which affects the response time of the system. The disadvantages of oxide heaters include high cost and brittleness, as well as objective difficulties with the transition to a flexible form factor.

Micro and nanowire films [5] are the most promising elemental base for transparent heaters. This type of coating allows combining high optical transparency and low surface resistance and, as a consequence, low energy consumption, as well as compatibility with a polymer carrier, which is the first requirement for transparent heaters. A number of studies have shown the possibility of realizing efficient transparent IR heaters based on the films of silver [6] and copper [7] nanowires, as well as micro and nanomeshes obtained by various methods of lithography [8–10]. However, the proposed methods are either expensive or difficult to scale, which prevents the emergence of industrial transparent heaters based on micro and nanowire films.

However, it is worth noting that the thermal and chemical stability is greatly reduced [11] in transition to micro and nano-scale. The given circumstance increases the relevance of the development of methods for passivating metallic micro and nanostructures.

This paper proposes the method of forming GO / Cu micromesh composite transparent conductive coatings. The micromesh coating is formed by vacuum deposition of copper on a self-organized template resulting from cracking of a gel film during the drying process and is a cheap analog of lithographic templates [12; 13].

The method of obtaining GO / Cu micromesh composite coatings. The first stage is the preparation of the substrate (polyethylene terephthalate, polyimide, glass, monocrystalline silicon), since the adhesion of the silica gel to the substrate is a key parameter that allows obtaining a system with controlled cracking morphology. The second stage is the application of a pre-prepared silica sol to the substrate. The third stage is the formation of a spatial gel network occurring due to evaporation of the solvent. At the same time there is a sharp increase in the viscosity of the solution; mechanical stresses leading to film cracking [14] are formed due to drying in the gel. The fourth stage of the process of obtaining micro-mesh coatings is metal sputtering on self-organized templates. At the fifth stage, selective liquid removal of silica cells with excess sprayed metal is performed. The difference in height between the surface of the template and the substrate allows selective removing the template with excess sprayed (sputtering) metal. As a result, only the micromesh coating remains on the substrate [12].

Silica sol was used to form a self-organized template. To obtain the sol, 3 ml of tetraethoxysilane $(C_2H_5O)_4Si$ and 1.5 ml of ethanol were mixed in one tube, and 0.01 ml of HCl was added to 1.5 ml of water in the second tube. Then the contents of the tubes were poured together and mixed vigorously. The sol was deposited on glass (2 mm) substrates with an area of 30 cm^2, using a Meyer rod (liquid film thickness of 25 μm) [13]. After applying the precursor, the films were dried in air for 15 minutes at a relative humidity of 35–40 %, which ensured complete cracking of the gel film. Layers of copper of 70, 140 and 210 nm thickness were deposited on the obtained templates by the magnetron method using a "Caroline D15" apparatus (ESTO-Vacuum, Russia). The choice of copper is due to its low resistivity and low cost.

The cost estimate of a micromesh coating is 1–3 Rub / m^2, while using silver as a micromesh material increases the cost of the coating by two orders of magnitude. After spraying metal, silica clusters are removed with adhesive tape.

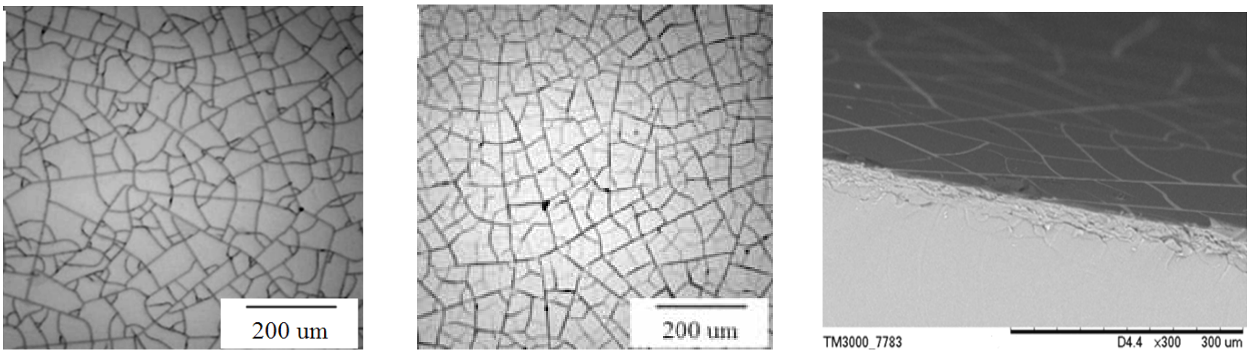
At the last stage, a graphene oxide film is deposited on the surface of micromesh using the spray-method. A 1.5 % aqueous solution of GO ("Acco Lab", Russia) prepared by Hammers method was used to create a protective film on metallic micromesh coatings. The film thickness was set by the volume of the solution applied per unit area. Two specific concentrations of GO were investigated: 0.01 ml / cm^2 and 0.05 ml / cm^2. After application, the system was dried in air at 70 ° C in order to remove water and seal the protective film.

The study of composite coatings by optical and electron microscopy. The obtained templates and copper micromeshes on glass substrates were studied by optical and electron microscopy. The microimage of the self-organized template is shown in fig. 1, *a*.

The average cell size obtained by statistical processing of 10 micrographs for this type of pattern is (43.3 ± 15.5) μm, the average crack width is (2.3 ± 0.6) μm. The metal fill factor of the surface is about (11.4 ± 1.5) %.

SEM images (images obtained with "Hitachi S-5500" electron microscope, KRSKC (Krasnoyarsk Regional

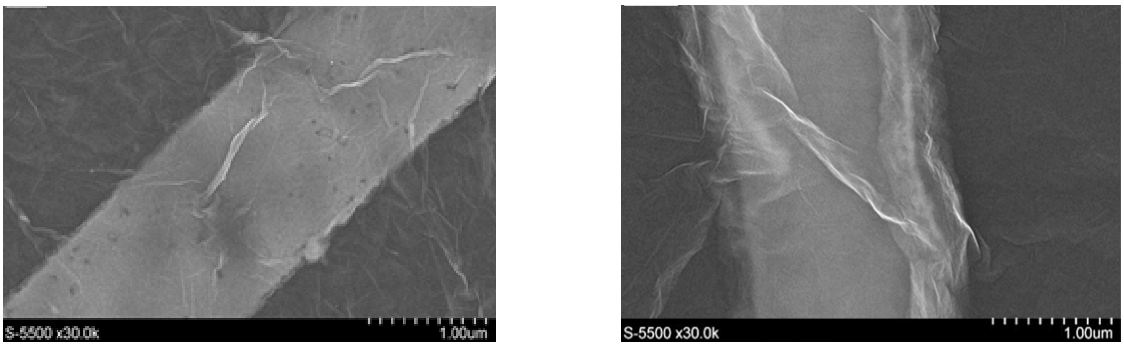
Shared Knowledge Centre) FIC KSC SB RAS) of a composite coating with different thickness of the graphene oxide film are shown in fig. 2, *a*, *b*. The microimage demonstrates high uniformity of the graphene oxide film. The protective layer of graphene oxide gives the coating a yellow-brown tint.



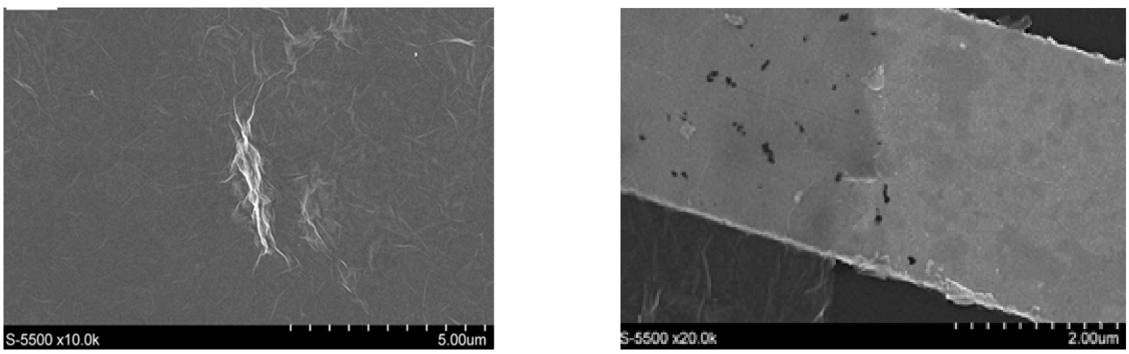
a *b* *c*

Fig. 1. A microimage of the silica template (*a*) and copper micromesh with a thickness of 70 nm (*b*), as well as the cross section of the micromesh on the glass (*c*)

Рис. 1. Микрофотография кремнезёмного шаблона (*а*) и медной микросетки толщиной 70 нм (*б*), а также скол микросетки на стекле (*в*)



a *b*



c *d*

Fig. 2. SEM-images of composite coatings of GO (0, 01 ml / cm^2) / Cu micromesh (*a*) and GO (0, 05 ml / cm^2) / Cu micromesh (*b*); the morphology of the graphene oxide film inside the micromesh cell (*c*); the boundary of the composite coating of GO (0,01 ml / cm^2) / Cu micromesh (*d*)

Рис. 2. СЭМ-изображения композиционных покрытий ОГ (0,01 мл/см^2) / Cu-микросетка (*а*) и ОГ (0,05 мл/см^2) / Cu-микросетка (*б*); морфология пленки оксида графена внутри ячейки микросетки (*в*); граница композиционного покрытия ОГ (0,01 мл/см^2) / Cu-микросетка (*г*)

The presence of folds characteristic of graphene materials is the evidence of the high-quality coating of the micromesh with a graphene oxide film and indicates its coherence.

The main advantage of the spray-method is the high homogeneity and continuity of the graphene oxide film over the entire area (fig. 2, *c*, *d*).

Optoelectronic properties of OG / Cu micromesh composite coatings. The spectral dependences of the optical transmittance of the composite coating at the main technological stages were measured in the range of 400–800 nm with the aid of Shimadzu UV-3600 spectrophotometer (KRSKC FIC KSC SB RAS) and are shown in fig. 3, *a*. The micromesh coating with a metallization thickness of 70 nm has a flat transmission spectrum in the entire considered range. Fresnel reflection from the substrate boundaries is about 9.5 % at the wavelength of 550 nm; the transmittance of the micromesh is 89.5 % without taking into account the influence of the substrate. The composite coating of GO (0.01 ml / cm^2) / Cu micromesh has uneven transmission, greatly losing transparency in the blue-violet spectral region due to the passivating layer of graphene oxide, which has an absorption peak in the UV region characteristic of aromatic compounds [15]. At the wavelength of 550 nm, the composite coating has a transparency of 86.8 % (minus Fresnel reflection from the substrate). The transparency of the graphene oxide layers at the wavelength of 550 nm was 97.3 % for a film of 0.01 ml / cm^2 and 90.2 % for a film of 0.05 ml / cm^2, respectively.

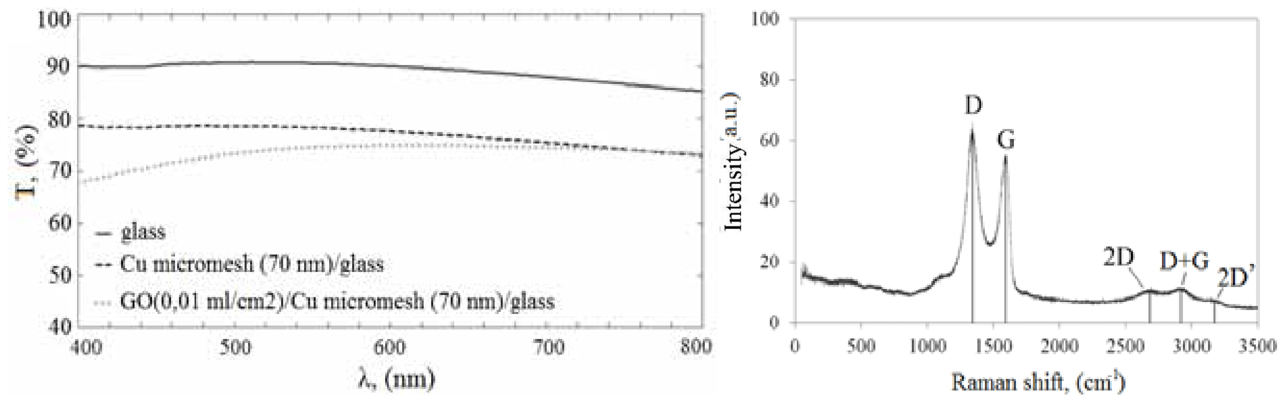
Fig. 3, *b* shows Raman spectrum of a graphene oxide film (measurements were performed on a HoribaJobinYvon T64000 spectrometer, KRSKC FIC KSC SB RAS). The spectrum of graphene oxide shows two main peaks characteristic of this system: the G-line, which characterizes the vibrations of the sp^2 carbon bond system (~1580 cm^{-1}) (graphite-like zone), and the 2D-line (~2700 cm^{-1}), which is an overtone of D-line (defective area) (~1330 cm^{-1}). The appearance of the D-line for samples of GO indicates the formation of a defective structure with respect to graphite. The appearance of a regular peak in the region of 2700 cm^{-1} indicates a small number of layers in the GO structure [16]. Overtones D + G and 2D′ are typical for graphene oxide, but they are not used for the qualitative characterization of graphene oxide films.

The effect of the thickness of magnetron sputtered copper and the thickness of the graphene oxide film on the optoelectronic properties of composite coatings are given in table.

Increasing the thickness of the metal threefold reduces the surface resistance of the coating while reducing transparency by 3.8 %; it is also one of the advantages of mesh microstructures.

Stability of composite coatings. The resistance of the coatings to oxidation by atmosperic oxygen was evaluated by the change in the specific resistance of the coatings during the test. As it is known from the literature, thin vacuum copper is slowly oxidized by atmospheric oxygen. The resistance of the copper mesh with the thickness of 70 nm at room temperature and humidity increased by 36 % for 30 days due to oxidation by oxygen in the air. A micromesh passivated by a graphene oxide film remained stable throughout the entire observation time (fig. 4, *a*). Moreover, in these conditions, the thickness of the GO film does not matter.

Fig. 4, *b* shows the stability of the coatings during their exposure at a temperature of 100 °C within 24 hours. The coating on the basis of copper micromesh without a passivating graphene oxide layer increased its resistance from 21.3 Ohm/sq to 3.2 kOhm/sq during the test, which resulted in a complete loss of functionality. The composite coating with a protective film of graphene oxide (0.01 ml / cm^2) increased its resistance by 7.6 % from 21.7 Ohm / sq to 23.3 Ohm / sq during the test. A slight degradation of the coating by the graphene oxide protective film is most likely due to the presence of a certain number of open tracks, which begin to oxidize during the heating process. A passivating graphene oxide layer with a specific concentration of 0.05 ml / cm^2 showed complete tightness during the entire observation period.



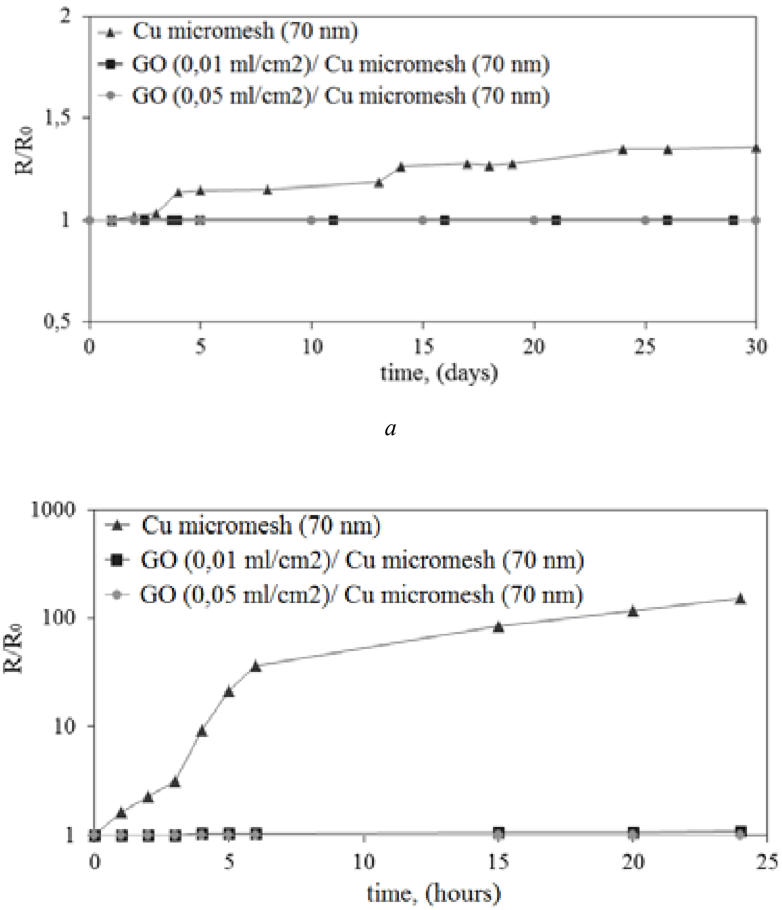
a *b*

Fig. 3. Spectral transmittance of the composite coating at the main technological stages (*a*); Raman spectrum of a thin graphene oxide film (*b*)

Рис. 3. Спектральное пропускание композиционного покрытия на основных технологических этапах (*а*); спектр комбинационного рассеяния тонкой пленки оксида графена (*б*)

The influence of technological parameters on the optoelectronic characteristics of composite coatings

Coating	h_{Cu}, nm	Rs, Ohm/sq	*T*(550 nm), %
Cu micromesh	70	21.3	89.5
Cu micromesh	140	14.2	87.9
Cu micromesh	210	8.6	85.7
GO (0.01 ml/cm^2) / Cu micromesh	140	15.3	85.1
GO (0.01 ml/cm^2) / Cu micromesh	210	8.9	82.8
GO (0.05 ml/cm^2) / Cu micromesh	140	15.5	77.2
ITO (commercial sample)	200	15	90



a

b

Fig. 4. Change in sheet resistance of coatings with exposure to air for a month (*a*) and daily exposure at 100 ° C (*b*)

Рис. 4. Изменение удельного поверхностного сопротивления покрытий при выдержке на воздухе в течение месяца (*а*) и суточной выдержке при 100 °С (*б*)

The study of a transparent heating element. The operation modes of the transparent heater were investigated on the GO system (0.05 ml / cm^2) / Cu micromesh (140 nm) / glass, the surface resistance of the coating was 15.3 Ohm / sq.

In the heater operation mode, a constant electric current was passed through the composite coating, heating it according to the Joule–Lenz law [17]

$$Q = I^2 R_s \Delta t . \qquad (1)$$

The heat released by the composite coating consists of three components, which can be written with consideration of Ohm's law as follows:

$$\frac{U^2}{R_s}\Delta t = Q_{cond} + Q_{conv} + Q_{rad} , \qquad (2)$$

where Q_{cond} is the summand responsible for the substrate heating, Q_{conv} is the convective summand, Q_{rad} is the summand responsible for radiant heat transfer [17].

Thermograms were measured using a Testo 875-2 thermal imager. Fig. 5, *a* shows a thermogram of the composite coating under investigation with a scattered power of 0.6 W/cm^2. The thermogram demonstrates the heating of high uniformity over the entire area of the coatings under study, in some areas there is a slight variation of 3–5 °C, which is most likely due to fluctuations of the surface resistance of the mesh coating. The heating rate of the system is due to the thermal conductivity and thickness of the substrate, as well as the heat transfer coefficient of the composite coating. The composite coating on glass has a response time of about 200 seconds (fig. 5, *b*), the same for all samples and determined by the substrate.

When the voltage of 15 V was applied, the specific power dissipated on the coating was 0.6 W / cm^2, as a result of which the coating heated to the temperature of 97.2 °C (fig. 5, *b*). The coating functioned within 24 hours without change in conductivity.

In general, it is worth noting that composite coatings on glass are able to work stably up to temperatures of about 150 °C (1 W/cm^2). Further increase in temperature above 200 °C leads to thermal degradation of the graphene oxide film. According to a thermogravimetric analysis carried out in [18], heating of graphene oxide in air above 200 °C is accompanied by its oxidation to gaseous products with loss of 20 % of weight, and the subsequent oxidation of the copper micromesh, which causes an increase in surface resistance by 50 % (fig. 5, *c*).

Fig. 6 shows the comparison of the effectiveness of a composite transparent heater with an ITO-based heater on a 2 mm thick glass substrate [17]. Since the substrates in experiments are the same, it is appropriate to compare their effectiveness.

The linear approximation of the experimental points obtained for the composite coating in the present work and for the ITO coating from [17] makes it possible to estimate the thermal resistance of heaters, measured in °C·cm^2·W^{-1}, and determining to what temperature the coating will heat up during power dissipation 1 W/cm^2. Approximation results give heat resistance values of 94.04 °C· cm^2·W^{-1} for ITO and 134.2 °C·cm^2·W^{-1} for composite coating, which is comparable to the best literary results.

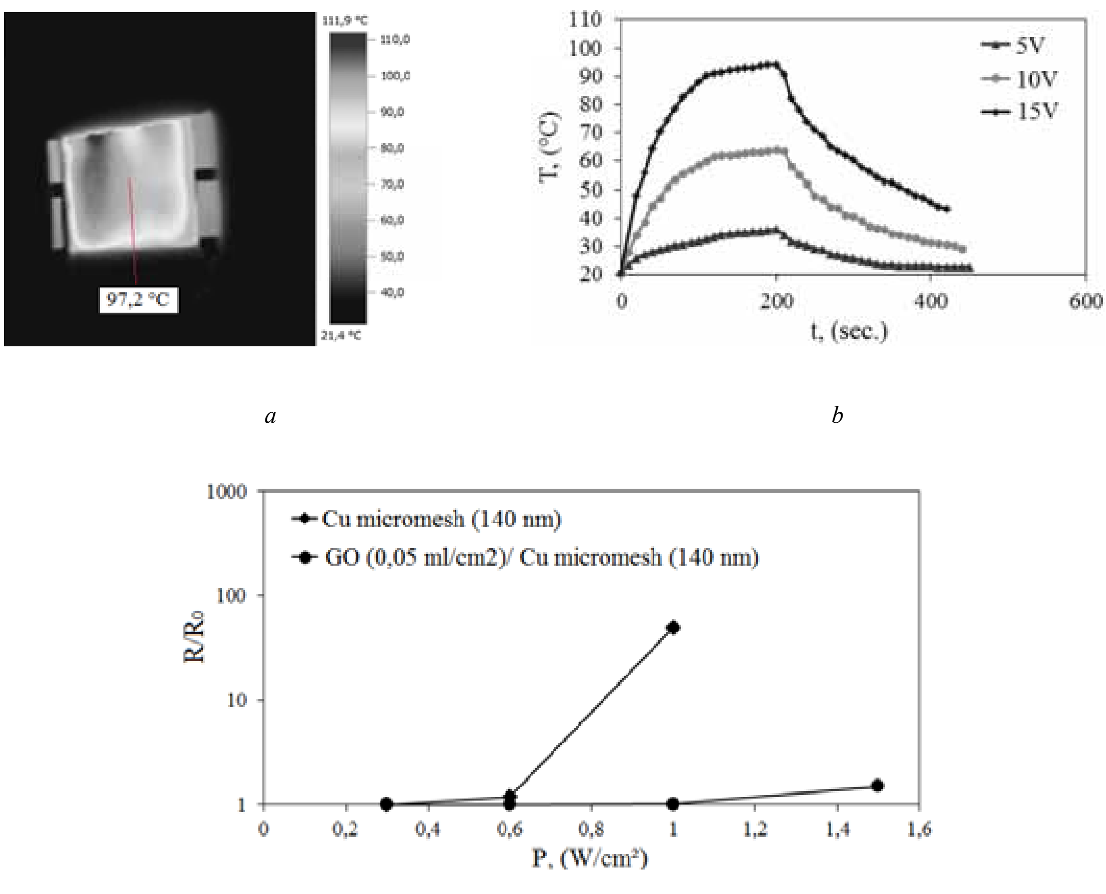


Fig. 5. Thermogram of the coating of the GO / Cu micromesh (140 nm) at the voltage of 15 V and dissipated power of 0,6 W/cm^2 (*a*); temperature profiles of the composite coating at the different dissipated power (*b*); the stability of coatings at critical values of the power dissipated (*c*)

Рис. 5. Термограмма покрытия ОГ / Cu-микросетка (140 нм) при напряжении 15 В и рассеиваемой мощности 0,6 Вт/см^2 (*а*); температурные профили композиционного покрытия при различной рассеиваемой мощности (*б*); стабильность покрытий при критических значениях рассеиваемой мощности (*в*)

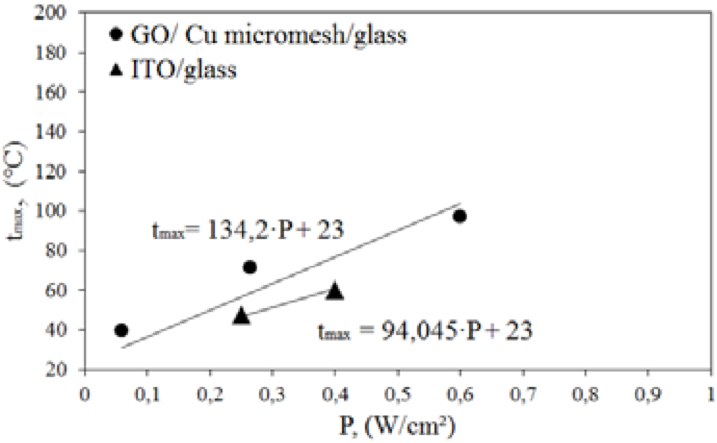


Fig. 6. Comparative efficiency of a composite coating with a literature ITO on an equivalent substrate [17]

Рис. 6. Сравнительная эффективность композиционного покрытия с литературным ITO на эквивалентной подложке [17]

The disadvantages of mesh coatings include extensive areas with low thermal conductivity (mesh cells), as a result of which certain areas of the coating can significantly overheat. In this context, it is interesting to consider the possibility of the reduction of graphene oxide having a thermal conductivity in thin films of > 5 W/m·K, which will allow a more even distribution of heat in space unoccupied by metal. An example of such a composite was proposed in [19].

Conclusion. The results of the study of a new approach to the formation of transparent heaters with enhanced performance characteristics with respect to classical solutions based on oxide films are presented. The composite coating is a copper micromesh, obtained using a self-organized template stabilized with graphene oxide. A composite coating with the surface resistance of 8.9 Ohm / sq was obtained with the transparency of 82.8 %.

High uniformity of heating and stability of the composite coating are shown when operating under heating up to 97.2 ° C for a long time (24 hours). Composite transparent heaters of GO / Cu micromesh demonstrate higher efficiency relative to common transparent heaters based on ITO. The composite GO / Cu micromesh coating on a glass substrate, 2 mm thick, is characterized by the thermal resistance value of 134.2 $°C·cm^2·W^{-1}$, while the ITO literary sample on an equivalent substrate is characterized by the thermal resistance of 94.04 $°C·cm^2·W^{-1}$. This fact opens up prospects for the use of the composite GO / Cu micromesh coating as a transparent anti-icing coating in aerospace industry.

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