

A thin-film polymer heating element with a continuous silver nanowires network embedded inside

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Abstract

This study presents a method for fabricating a film-based heating element using a polymer material with an array of intersecting conductive elements embedded within it. Track-etched membranes (TM) with a thickness of 10 μm were used as the template, and their pores were filled with metal, forming a three-dimensional grid. Due to the unique manufacturing process of TM, the pores inside intersect with each other, allowing for contacts between individual nanowires (NWs) when filled with metal. Experimental results demonstrated that filling the TM pores with silver allows for heating temperatures up to 78 degrees without deformation or damage to the heating element. The resulting flexible heating element can be utilized in medical devices for heating purposes or as a thermal barrier coating.

Supplementary material for this article is available [online](#)

Keywords: track-etched membranes, template synthesis, nanowires, film heater

(Some figures may appear in colour only in the online journal)

1. Introduction

Currently, the demand for various flexible electronic components such as conductors, sensors, and active elements remains high [1–6]. In addition to their use as conductors for low currents, these elements can also serve as localized heaters. When currents in the range of several tens of milliamperes pass through a network of nanowires (NWs), the surface of the film begins to heat up. The temperature is typically limited by the thermal stability of the polymer substrate. The characteristics of silver NWs have expanded their applications from transparent conductive electrodes to strain sensors, light-emitting devices, displays, heaters, and other flexible devices. It is expected that research in the field

of flexible/wearable electronics will become increasingly in demand as the wearable electronics market continues to grow.

Transparent electrodes are of particular interest as they can serve as heaters for anti-fogging systems (such as glasses and screens) or in medical devices for heating, thermal barrier coatings, optical elements, or electronic components [7–9]. With the advancements in nanomaterial synthesis techniques, the fabrication of flexible composite heaters, particularly film-based ones, has become feasible. It is envisaged that such heaters can be employed in various optoelectronic interface devices, including touchscreens, solar cells, light-emitting devices, to maintain their temperature regime [10–12].

A separate interest lies in biocompatible devices that can be attached to the human body. It is possible to create a

heating ‘pad’ for the skin, which can be used for localized heating. These ‘pads’ are fabricated based on a composite of polymer with metallic nanowires using the cold-pressing method [13].

Over the past two decades, indium tin oxide (ITO) has been widely used as a transparent conducting electrode in GaN-based light-emitting diodes (LEDs) due to its high optical transmittance (approximately 90% at 450 nm) and low sheet resistance (typically 0–3 ohms square⁻¹ with a thickness of 150 nm) [14, 15]. However, the application of ITO in LEDs is limited due to factors such as the high material cost associated with the depletion of indium and its sensitivity to acidic and alkaline chemical sources [4].

A modern approach to addressing this issue is the transition from electrode sputtering to the deposition of metal NWs such as silver or copper [12, 14], or carbon nanotubes [15]. One-dimensional materials such as graphene can also be used [16]. NWs are promising as a solution for replacing existing conductive materials such as indium tin oxide (ITO) and conductive polymers [17]. Compared to ITO films, films with a network of silver NWs exhibit better transparency and the ability to manufacture heaters using roll technology [8, 18]. In one of the early works [11], it was proposed to fabricate heaters using silver nanowires on the surface of glass or a polymer film made of polyethylene terephthalate with a thickness of 1.3 μm . Metallic NWs can be applied to various substrates and integrated into the film [19] through pressing or by creating multilayer films.

Metal NWs possess excellent electrical properties and superior mechanical strength under repeated mechanical stress, making them a promising material for practical transparent flexible/wearable devices. Ag NWs exhibit good electrical conductivity and high optical transparency, making them commonly used as transparent conductive electrodes [9]. Such systems with NWs can also be used as flexible heaters [18]. The key characteristics of these film heaters include good stretchability, flexibility, high strength, a wide temperature range, fast heating rates, and chemical resistance of the film. There are various methods for fabricating thin-film thermoelements, most of which involve integrating silver nanowires onto polymer films.

There have been several studies in which transparent conductive electrodes were obtained by integrating silver nanowires with a diameter of 35 nm and graphene as a protective layer. The silver was deposited using the spin-coating method, which has a disadvantage of non-uniform deposition due to agglomeration, resulting in areas with low resistance where high currents and excessive surface heating may occur [20]. As a result of such chaotic deposition of conductive structures, a net of copper or silver wires is formed on the surface of the polymer film [10]. The optical transmittance coefficient of flexible heaters based on nanowires has reached 86.4% in certain studies [21].

Transparent heating films are of particular interest. The possibility of achieving maximum transparency and stability with easy scalability is being investigated. In some cases, nanowire nets are introduced into specially prepared fabrics [22] or in the form of multilayer films [23] for thermotherapy

applications [7, 24, 25]. A study [26] analyzes different approaches to fabricating thin film heaters. Specifically, it is mentioned that only nanowires with a large diameter are suitable. As the diameter decreases, problems arise with the contact between neighboring nanowires. Additionally, for certain applications such as electronics, the film surface needs to be smooth, which may not always be achievable when using a nanowire net, especially with a large diameter. Various methods are employed to address the contact issue between individual NWs, such as thermal annealing [12], pressing [12, 13], electron beam irradiation [27, 28], pulsed ion beams [27], or passing a high current through the net formed by nanowires [11].

The most commonly used method for depositing nanowires on surfaces is spin coating. In addition to spin coating, nanowires can also be applied using a brush followed by annealing or brief heating with an ion beam or optical radiation using a lamp to fuse the nanowires together and form a continuous net [13, 27, 29].

It is also possible to create a NWs net using drop application: a solution of dispersed NWs is dropped onto a substrate, and the residual solvent is evaporated [29]. The optical transparency of the cast NWs net largely depends on the properties of the solution, such as the concentration of NWs or the rate of solvent evaporation.

The process of depositing NWs onto large surfaces can be achieved using spray coating technology [11, 30, 31]. This approach allows for the uniform coverage of large areas with silver NWs on various substrates. Additionally, spray coating is a suitable method for depositing multiple layers of different materials [31, 32]. In the study [33], for the creation of a heating element on surfaces of complex and large dimensions, a crackling lacquer is applied with the filling of cracks by metal (Ag). The stability of the heating over time for silver nanowire nets can range from 2 to 30 h under different voltage values [11, 30].

It is worth noting that the surface roughness of any thin film is an important quality factor for subsequent device fabrication and integration [34]. The ideal situation is to have as smooth a thin film as possible. For example, percolated films with silver NWs exhibit high surface roughness due to the large diameter of the NWs (typically ranging from 20 to 100 nm), which poses a significant challenge for the deposition of active layers in solar cells or light-emitting devices. As a result, flexible optoelectronic devices based on silver NWs typically have inferior performance compared to standard ITO-based films, as these nanowire-based structures contain a large number of voids, making charge transport highly inefficient [35].

This problem can be addressed by integrating the NWs inside the polymer film. In our work, we propose fabricating a film-based polymer heater using the template synthesis method of metallic NWs based on a track-etched membrane (TM). The TM is a polymer film with a thickness of 10–12 μm that contains a system of cylindrical channels of a specific diameter, which are created through the irradiation of the initial polymer film with heavy ions followed by subsequent physicochemical processing. The use of TM as a template

makes it possible to controllably produce nanowires from various metals with unique electrical, optical and magnetic properties [36–38]. Specifically, this technology allows the fabrication of polymer films with a three-dimensional network of interconnected NWs embedded within the polymer [39]. In previous studies [40–43], successful observation of thermoelectric and magneto-resistive effects in such systems has been reported.

By selecting a TM with a high number of pore intersections, a high electrical conductivity can be achieved in the resulting film due to the increased number of NWs intersections within the heater structure. The estimation of the number of pore intersections within the thickness of the TM can be done theoretically and controlled by the conditions of irradiation with accelerated ions. Additionally, the presence of intersecting pores can be observed in the prepared TM using the methodology proposed in [44].

The heating element obtained through the chemical dissolution of the metallic contact layer can be transparent if the density of the TM channels is low. The high elasticity of the polymer heater is achieved because the metallic NWs occupy 5% or less of the volume of the track-etched membrane. Our main advantage lies in the use of an original synthesis method for the electrochemical growth of a silver nanowire mesh within the polymer, enabling the flexible heater to operate up to the point of complete rupture. Also, this heater exhibits good chemical and mechanical stability and offers several technological advantages over existing alternatives, such as low production cost, heater flexibility, high chemical and mechanical resistance, uniform heat distribution across the heater's surface, low surface roughness, and smooth heating due to the low thermal conductivity of the polymer. Thus, flexible heaters manufactured using the original synthesis method described in this paper have prospects not only for use in mass production but also for the potential development of transparent flexible heaters by selecting a porous membrane with low pore density. The track etching technology allows fabrication of membranes the porosity of which lies in a very wide range.

2. Materials and methods

2.1. Track-etched membranes

The track-etched membrane was fabricated from a biaxially oriented polyethylene terephthalate (PET) film with the thickness of 10 μm . The film was irradiated with accelerated ^{136}Xe ions on the U-300 cyclotron of the Flerov Laboratory of Nuclear Reactions. The ions entered the film under different angles within the interval of $\pm 30^\circ$ to normal (see figure 1). The irradiation was performed through an array of ~ 0.5 mm stainless steel wires to produce shadows on the film, which reinforce the resulting membrane. Using mild etching (1 M NaOH, 80 $^\circ\text{C}$), the ion tracks were transformed into cylindrical channels 100 nm in diameter [45]. The surface density of the channels was $1.2 \times 10^9 \text{ cm}^{-2}$.

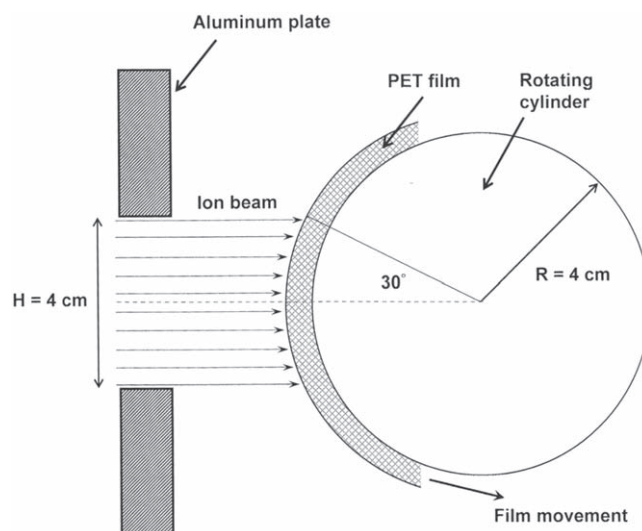


Figure 1. Schematic of the irradiation mode which provided generation of an array of mutually intersecting pore channels [46]. The film circumflexes a horizontal cylindrical shaft 4 cm in radius. The ion beam, homogeneously spread in both vertical and horizontal directions, impinges the film through a window 4 cm high.

2.2. Microscopy

After the synthesis, control samples were detached from the polymer template and examined using scanning electron microscopy (SEM) using a JEOL JCM-6000 instrument (Japan).

2.3. Measurement of heating

To measure the heating effect, a setup consisting of a laboratory power supply and a thermal imaging camera (UNIT UTi260B, China) was used. The thermal imaging camera was employed to monitor and ensure the uniformity of heating. To focus on the sample and observe magnified images of individual membrane fragments, a magnifying lens was used in conjunction with the thermal imaging camera. This allowed for detailed examination of the heating distribution and temperature variations across the sample.

2.4. Measurement of the deformation-strength characteristics

The deformation-strength characteristics in the mode of uniaxial tension of the original TM and TM with silver-filled pores were studied on an Autograph AGS-10 kN tensile testing machine by Shimadzu (Japan). The stretching rate was of 4 mm min^{-1} . The samples were strips $5 \times 50 \text{ mm}$ in size. Tensile tests were carried out with parallel measurement of electrical resistance using a Unit UT-612LCR bridge impedance meter. The resistance measurement error did not exceed 3%.

3. Results and discussions

3.1. Fabrication of the Heater

In the first stage, contact pads made of silver with a thickness of 50 nm were deposited onto one of the surfaces of the track-etched membrane (figure 2(a)) using a vacuum deposition

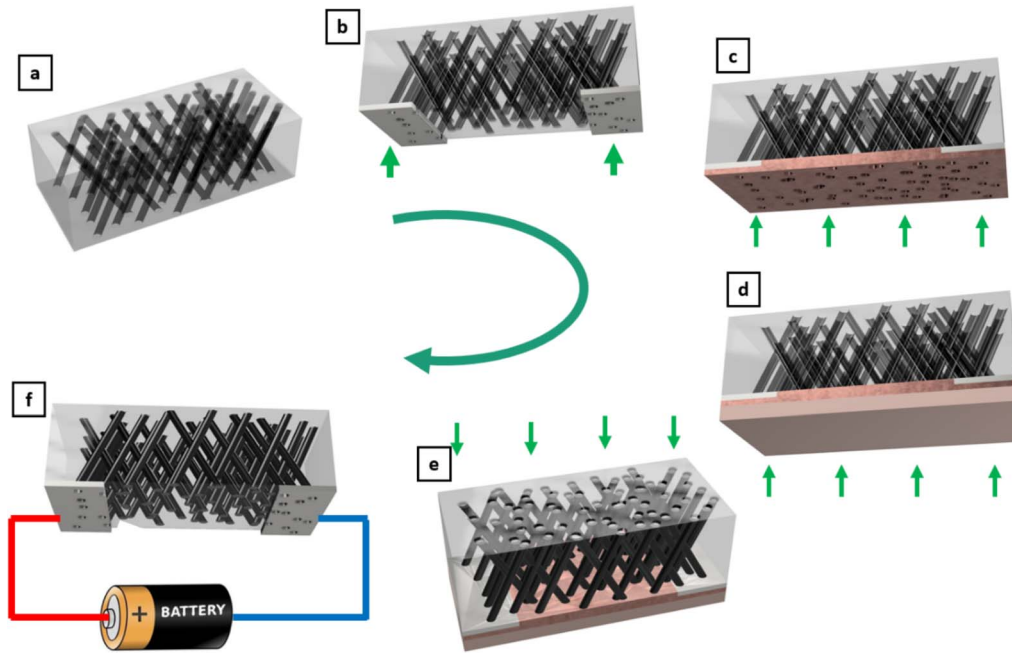


Figure 2. Scheme of synthesis of a thin-film polymer heating element with a 3D array of silver NWs inside the pores of TM.

technique. The vacuum deposition was performed using a resistive evaporation method on a SAHA universal vacuum system (Danaeng, Russia). Subsequently, a copper layer with a thickness of 50 nm was also deposited onto the entire surface of the TM, including over the contact pads, using the resistive evaporation method (figure 1(c)) to create a continuous conductive path.

Then, on top of the previous deposition, a copper layer with a thickness of several micrometers was electrochemically plated to seal the pores and reinforce the thin deposited layer (figure 2(d)). The electrodeposition was carried out in a galvanostatic mode using a copper electrolyte with the following composition: $\text{CuSO}_4 \cdot 7\text{H}_2\text{O}$ -300 g l^{-1} ; H_2SO_4 -15 g l^{-1} .

In the next step, a special electrochemical cell was used to deposit silver into the pores of the TM (figure 2(e)). The Ellins P-2X potentiostat–galvanostat (Russia) was employed as the current source. The electrodeposition was carried out in a galvanostatic mode with a current density of 2.5–5 mA cm^{-2} using an electrolyte with the following composition: AgNO_3 -40–47 g l^{-1} , $\text{K}_3[\text{Fe}(\text{CN})_6]$ -40–60 g l^{-1} , K_2CO_3 -35–40 g l^{-1} , KSCN -80–120 g l^{-1} . The deposition process was stopped once the pores were completely filled, which was determined by a decrease in voltage across the galvanic cell.

In the final step, copper was selectively dissolved from the sample surface using a diluted 1:2 solution with the following composition: H_2O_2 (3%)-1 l; citric acid ($\text{C}_6\text{H}_8\text{O}_7$)-300 g l^{-1} ; NaCl -50 g l^{-1} (figure 1(f)). After complete dissolution of copper, the samples were rinsed with distilled water, dried, and applied contact with conductive silver paint.

3.2. Microscopy

For SEM after synthesis, the structure of the NWs inside the pores of TM was investigated. For this purpose, the TM was

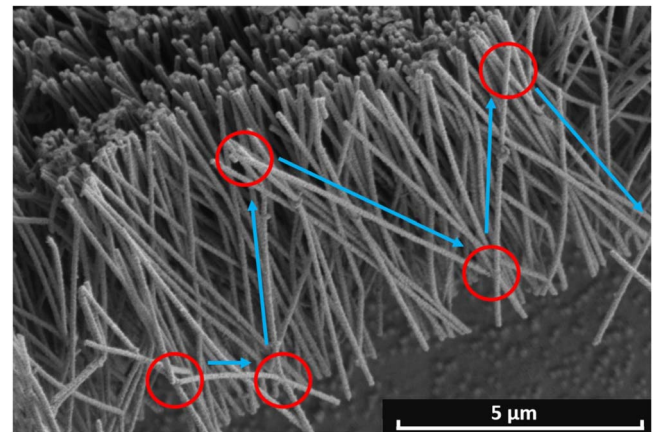


Figure 3. Micrographs of a network of silver NWs with a diameter of 100 nm after removal of the polymer TM. Intersections of nanowires are marked with red circles. Blue arrows indicate electrical circuit. Reproduced from [47]. CC BY 3.0.

dissolved in an alkaline solution, and the resulting nanowire network was examined using scanning electron microscopy. Figure 3 shows the microscopy results. It is evident that the cross-intersections of the pores inside the polymer contribute to the formation of a continuous network of silver NWs.

3.3. Thermometry

A samples with dimensions of 75 × 28 mm were fabricated. The active area size was of 48 × 28 mm. The appearance of the 3rd sample from both sides is shown in figure 4. The resistance of the sample is 6 Ohms. Temperature measurements were conducted during heating at different voltages ranging from 1 to 4.5 V. The temperature and uniformity of heating were monitored using a thermal imaging camera. The

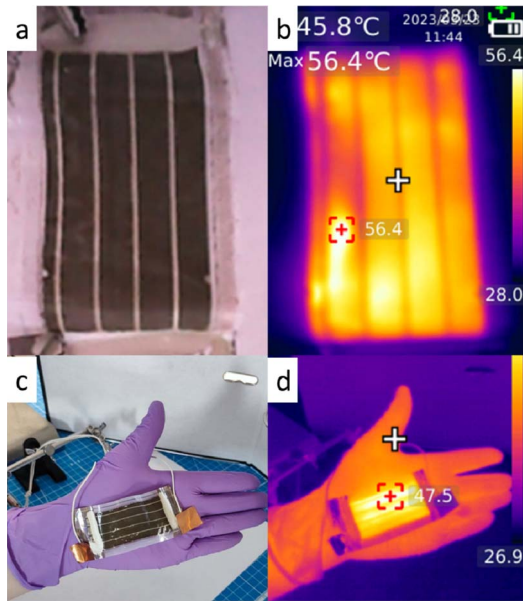


Figure 4. The appearance of the sample and thermograms at different voltages.

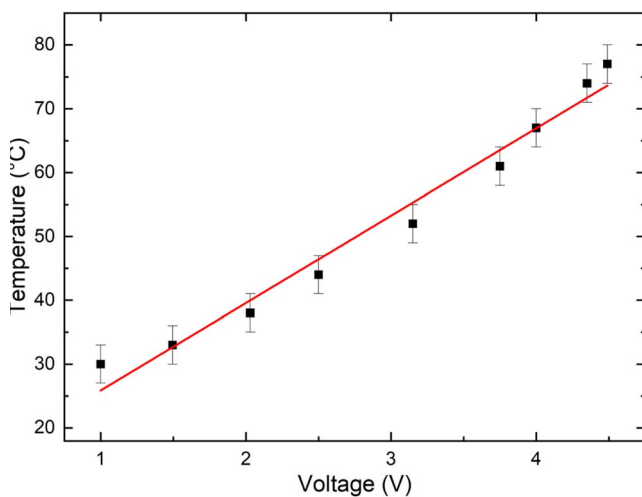


Figure 5. Voltage dependence on temperature for a series of samples.

maximum temperature reached at a voltage of 4.5 V was 78 °C.

To collect statistics, heating temperature was measured from electrical characteristics for a series of three samples. The dependence of temperature on voltage consumption revealed a linear temperature increase within the range of 30 °C–78 °C (figure 5). Heating was carried out up to 78 °C, which was achieved at a current of 420 mA. In this case, the power consumption reached the value of 1.8 W. Heating to higher temperatures was not conducted to avoid exceeding the glass transition temperature of the polymer matrix made of PET, which is 82 °C [48].

Separately, measurements were carried out to determine the stability of the heater over time. The measurements were carried out cyclically turning on and off the current on the sample. The current on the sample was 420 mA for 20 s, and the off time was also 20 s. The heating temperature at the

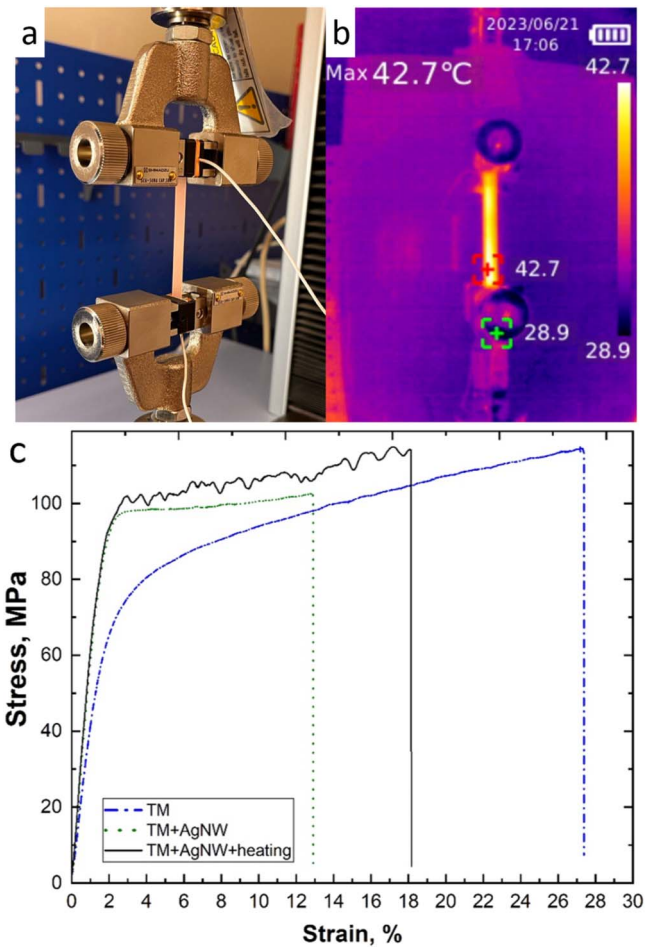


Figure 6. Deformation dependences for track-etched membranes and track-etched membranes with silver-filled pores.

Table 1. Mechanical characteristics of the investigated samples.

	Tensile strength, MPa	Failure strain, %	Elastic modulus, GPa
TM	113 ± 2	29 ± 1	3,9 ± 0,1
TM + Ag NW	100 ± 10	14 ± 4	5,7 ± 0,7
TM + Ag NW + heating	104 ± 16	15 ± 17	6,2 ± 0,6

beginning of measurements and after 4 h of measurements was 78 °C, with a slight temperature change of several degrees during the measurement process, apparently due to instability of the ambient temperature.

To determine the maximum operating temperature, test measurements of the maximum heating temperature were carried out. To do this, a voltage of 100 mA was applied to the heater sample from a current source with a stepwise increase of 3 mA every 10 s. As a result, the destruction of the film occurred at a temperature of 160 degrees, a voltage of 12.6 V and a current of 600 mA (see suppl. mater. 1).

3.4. Mechanical properties of the heater

The measurement results are presented in table 1. Each value represents the average data from at least 5 measurements.

The strength of the composites is lower than that of the TM, which is attributed to the contribution of the metal in the stretching process due to the low adhesion of silver to the polymer. Additionally, the metal in the TM pores hinders the increase in strength during stretching by evolving pore shape and reducing stress concentration, as observed in the stretching of TM. The elastic modulus of the composite is 30% higher than the elastic modulus of the TM matrix due to the volume fraction of NWs and the interaction of their elastic fields. The region of the stress–strain curve that characterizes linear-elastic deformation ends at $\varepsilon = 3\%$.

The specific conductivity during sample stretching remains nearly unchanged until failure and amounts to $0.03 \cdot 10^6 \text{ Sm m}^{-1}$ (compared to the value of $63 \cdot 10^6 \text{ Sm m}^{-1}$ for bulk metallic silver [49]).

Figure 6 illustrates the process of measuring the failure strain with heating. The uniform heating of the entire sample surface is clearly visible. Figure 6 shows the stress–strain curves for a TM and a TM with silver-filled pores. The stress–strain diagram (σ – ε) for the TM with silver-filled pores during the plastic deformation stage exhibits a nonuniform pattern as the stationarity of the destruction process is violated. This is likely due to the occurrence of local overheating in the areas where the NWs intersect, leading to localized changes in strength.

4. Conclusion

In conclusion, this study presented a methodology for synthesizing three-dimensional networks of NWs within the pores of TM. A technology for uniform electrodeposition of NWs on large-area templates using a reversible deposition mode was developed. Additionally, a method for selectively removing the auxiliary copper layer from the samples was suggested. The heating process of the metal NWs/polymer composite was demonstrated. The conductivity is maintained until complete failure. The maximum deformation magnitude is 15% for the heater. A temperature of 78 °C was achieved at a current of 420 mA and the power consumption reached the value 1.8 W for the active area size was of 48 x 28 mm. The linear dependency of temperature on voltage ensures the reproducibility of the results. Mechanical tests have shown that the film heater retains its functionality up to the moment of complete destruction with deformation of about 15%. When using a polymer matrix made of a more heat-resistant polymer, such as polycarbonate or polyimide, the technology will make it possible to produce a heater that can maintain temperatures above 100 °C.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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