Ultra-thin foldable transparent electrodes composed of stacked silver nanowires embedded in polydimethylsiloxane

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Abstract
We have prepared an ultra-thin flexible transparent conductive electrode with high folding endurance composed of randomly arranged silver nanowires (AgNWs) embedded in polydimethylsiloxane (PDMS). A simple preparation method was performed to connect a glass substrate coated with a AgNW network and a glass substrate coated with PDMS. The glass substrate was then removed after the PDMS solidified, and the AgNW–PDMS composite film was peeled off. Moreover, the problem of the high contact resistance caused by the random arrangement of AgNWs was solved by the local joule heat generated by applying voltage to both sides of the AgNW–PDMS composite structure to weld the overlapping AgNWs. The sheet resistance \( R_s \) of AgNW–PDMS composite films with different AgNW deposition concentrations decreased by 46.4%–75.8% through this electro-sintering treatment. The embedded structure of the AgNW–PDMS composite ensures better voltage resistance and environmental stability under high temperature and humidity conditions compared with a AgNW network attached to a glass substrate. Additionally, the substrate-free, excellent elasticity and high resilience characteristics resulted in the \( R_s \) value of the same composite electrode only increasing by 2.9 ohm sq\(^{-1}\) after folding four times. The advantage of the metal thermal conductivity makes the joule heat generated by electric injection rapidly diffuse and dissipate in the AgNW-based transparent heater with faster response time and smaller voltage drive than indium tin oxide.

1. Introduction

With the development of new materials and technologies in the field of electronics, the demand for multifunction and multi-application devices and electronic components has gradually increased. In particular, the recent launch of foldable display terminals has inspired and given more attention to development of truly foldable and rolled display devices. For these devices, preparation and performance optimization of transparent electrodes that can be flexibly formed are important steps [1]. The quality of the transparent electrode materials is the key factor to determine the performance of electron transport and collection in various photoelectric devices, including wearable electronics, light-emitting diodes, solar cells, touch screens, electronic paper and electromagnetic interference shields [2–5]. At present, market-oriented transparent electrodes are mainly metal oxide materials represented by indium tin oxide (ITO), which is widely used as the window electrodes in different photoelectric devices because of its excellent electrical conductivity and transmittance. However, the brittleness of ITO and the requirements of high-temperature annealing and a vacuum deposition environment are not compatible with potential flexible processing technology [6–9]. To solve this problem, several alternative materials have been developed by different research groups, such as graphene [10–12], carbon nanotubes [13–15], conductive polymers [16–18] and metal nanowires [1, 19–21]. Among these alternative materials that can be flexibly processed, metal nanowire (NW) structures, especially silver NW (AgNW) networks, have high...
optical transmittance and good conductivity properties. With the improvement of AgNW synthesis and coating technologies [22, 23], flexible AgNW-based transparent conductive structures with high performance can be prepared by a simple process on a large scale. The high conductivity of the metal itself combined with the network structure can enhance the carrier transport ability, which would be beneficial for improving the carrier injection and collection of AgNW-based electrodes in high-performance photoelectric devices [24]. And the versatility of AgNW-based networks as electrodes has also attracted some attention in the electrochemical applications [25–27].

However, many properties of AgNW networks still have a lot of room for improvement, such as the large contact resistance generated in the random overlap process, the poor mechanical stability caused by the weak contact between the AgNWs and the substrate, and the poor environmental stability of AgNWs [28]. To solve the problem of the high contact resistance, the contact area at the intersections in the AgNW network should be increased to remove the influence of the high contact resistance on carrier transport. Annealing treatment and an optical treatment process can be used to perform NW–NW fusion to achieve close contact [29–31]. High-temperature annealing or pulse-laser sintering can effectively weld the AgNWs together, but each process has an effect on flexible substrates. The local joule heat generated by applying voltage to both ends of the AgNW network can effectively produce the electro-sintering effect at the intersections of the AgNWs, which is an attractive methodology to address this issue. In view of the problems of the poor adhesion between the AgNWs and substrate and the lack environmental stability of the nanostructures themselves, coating layers of AgNWs can be constructed to improve the ability to resist external stress and prevent aggregation of surface defect states, thus achieving improvement of the mechanical stability and environmental stability. The hybrid materials introduced to protect AgNW network structures include graphene [32, 33], metal oxides [34, 35] and polymers [36, 37]. With the rapid development of organic electronic devices, the composite structure of AgNW networks and polymers is conducive to compatibility with flexible organic devices, and it is also beneficial to avoid the complex preparation process and vacuum preparation environment of graphene and metal oxides, respectively. Among these composites, it has been reported that the composite structure of a AgNW network and polydimethylsiloxane (PDMS) can effectively improve the stability of AgNWs [38, 39]. However, these composite structures still rely on substrates. This means that it is difficult for the transparent electrode to achieve arbitrary folding and for the integrated device to form a shape-preserving attachment to amorphous objects, such as skin, and thus they are not suitable for wearable devices and skin electronics. Therefore, a simple and flexible AgNW–polymer composite structure without a substrate support is required.

Here, we report an ultra-thin transparent electrode structure composed of a AgNW network embedded in PDMS. This flexible and free-form structure can effectively enhance the functionality of electrodes and make the constructed electronic devices no longer restricted to a planar surface. The local joule heat generated by applying voltage can effectively weld the overlapping positions of the AgNWs and solve the contact resistance problem of AgNW–PDMS composite structures under flexible machining. In contrast, the traditional sintering process is not simple and cannot be performed in a short time. Owing to the excellent hydrophobicity of PDMS, the AgNW-embedded composite structure shows good resistance to a high-temperature and high-humidity environment. Owing to the close contact with the PDMS polymer and formation of a shape-preserving coating, the AgNW-network electrode shows better voltage resistance in the electro-sintering process than that of a AgNW-network deposited on a substrate. The ultra-thin hybrid structure (total thickness of ∼192 μm) ensures its stretching and warping ability, and the sheet resistance ($R_s$) only increases by 2.9 ohm sq$^{-1}$ after the AgNW–PDMS composite is folded in half four times. This excellent flexibility enables it to be used as an electrode of skin electronics or as an electric heater at joints. In contrast to metal oxides, the excellent electrical and thermal conductivity of Ag make the temperature of the AgNW–PDMS composite electrode increase from room temperature to 70 °C at 5 V for 10 s.

2. Experimental

2.1. Fabrication of composite electrodes

A schematic of preparation of the ultra-thin AgNW–PDMS composite structure is shown in figure 1. Using a suspension of 0.5 mg ml$^{-1}$ AgNWs in dilute anhydrous ethanol, commercial AgNWs with average length of 10 μm and diameter of 80 nm were deposited on glass substrate 1 with length and width of 25 mm by the drop coating method to form a randomly arranged AgNW network. The deposition density of the AgNWs was changed by controlling the number of drops coated on the glass substrate. The commercial PDMS precursor (10:1 mixture of PDMS monomer:curing agent) was placed in a vacuum dryer for 10 min to remove the bubbles that formed during the mixing process. A drop of 150 μl of the PDMS precursor solution was then deposited on glass substrate 2 with length and width of 25 mm. Subsequently, the AgNW network deposited on glass substrate 1 was inverted on the glass substrate with PDMS droplets. The AgNW network was contacted and composited
with PDMS by the weight of the substrate 1 itself, and the whole structure was placed on a hot plate at 80 °C for 30 min to cure the AgNW–PDMS composite structure. Finally, glass substrate 1 was removed, and the AgNW–PDMS composite film was peeled from glass substrate 2 to form an ultra-thin (total thickness of ∼192 μm, as shown in figure S1(available online at stacks.iop.org/MRX/9/015006/mmedia)) and arbitrarily foldable transparent electrode based on a AgNW network without an attached substrate.

2.2. Characterization

The transmittance spectra of AgNW networks were measured with an ultraviolet–visible spectrophotometer (UV-2600, Shimadzu). Electrical characterizations were measured by the standard four-point probe method using a Keithley 6220 precision current source, a Keithley 2182 A nanovoltmeter, and a multi-range DC power supply (PSW 160–14.4, Gwinstek). The morphology and microstructure of AgNWs–PDMS composites were investigated by a field emission-scanning electron microscope (FE-SEM, JSM-7610F). The Electric heating performance was measured by the thermal infrared imager (E8, FLIR). The high temperature and high humidity aging environment were controlled by constant temperature and humidity chamber (LK 80 G, Sailham). The cyclic voltammetry performance was evaluated on CHI 760E (CH Instruments Ins.) electrochemical workstation.

3. Results and discussion

The transmittance and $R_s$ value are a pair of contradictory properties in the field of transparent electrodes, and we balanced these two photoelectric properties by regulating the deposition density of the AgNW network. The ultraviolet–visible transmission spectra of pristine AgNW networks with different deposition densities of AgNWs and their corresponding $R_s$ values are shown in figure 2. The overall transmittance spectra revealed that the AgNW networks showed excellent broad spectrum optical transparency, which plays an important role in enhancing the efficiency of light absorption or emission as a window electrode. The transmittance decreased with increasing deposition density of AgNWs (figure 2(a)). The correlations among the deposition density, $R_s$ value, and transmittance at 550 nm wavelength of the AgNW networks are shown in figure 2(b). When the deposition densities of AgNWs were 240 and 320 mg m$^{-2}$, the corresponding transmittance at 550 nm values were 84% and 72%, and the associated $R_s$ values of the AgNW networks were 368.7 and 35.6 ohm sq$^{-1}$, respectively. The results showed that the weak contact of the AgNWs led to high contact resistance in the process of random arrangement, and a high $R_s$ value is not conducive to electron migration in the network structure. Therefore, an effective treatment is needed to solve the problem of the high contact resistance in the AgNW–PDMS composite structure.

To obtain flexible composite structures containing organic polymers, the electro-sintering process was used to increase the contact area of the overlapping points of the AgNWs by utilizing the local joule heat in the network to eliminate the obstacle of electron diffusion caused by high contact resistance. The electro-sintering
performance was assessed by measuring the sheet resistance at different sintering voltages (figure 3). Different electro-sintering voltages were applied to both ends of the AgNW–PDMS composite electrode with deposition density of 240 mg m$^{-2}$. The ratio of the interelectrode resistance (electrode spacing 10 mm) after and before treatment significantly decreased with increasing electro-sintering voltage, as shown in figure 3(a). The interelectrode resistance of the AgNW-network composite electrode was effectively reduced from 506.8 to 51.8 ohm at 6 V sintering voltage for 2 min (see the insert of figure 3(a)). Similarly, electro-sintering treatment effectively reduced the influence of the contact resistance on electron transport for two other AgNW networks with deposition densities of 320 mg m$^{-2}$ (figure 3(b)) and 400 mg m$^{-2}$ (figure 3(c)). It was found that the AgNW–PDMS composite electrodes with lower resistance needed smaller sintering voltages to generate sufficient joule heat and weld the overlapping points of the AgNWs in the network. In addition to the
inter electrode resistance, comparison of the \( R_s \) values of AgNW networks with different deposition densities under the optimum sintering voltage is shown in figure 3(d). After electro-sintering treatment, the \( R_s \) values of the composite electrodes with AgNW deposition densities of 240, 320 and 400 mg m\(^{-2}\) decreased from 368.6, 35.6 and 13.6 ohm to 82.9, 12.6 and 7.2 ohm, respectively. This simple process that is suitable for flexible films was proven to be effective for improving the electrical properties of the AgNW–PDMS composite electrode. However, high sintering voltage was not conducive to optimization of the resistance, leading to an increase in the turning point of the \( R_s \) values of AgNW–PDMS electrodes with different deposition densities.

To determine the reasons for the sudden increases of the \( R_s \) values of AgNW–PDMS electrodes with different deposition densities, scanning electron microscopy (SEM) images of the composite electrode with deposition density of 320 mg m\(^{-2}\) before and after electro-sintering treatment were recorded (figure 4). As shown in figure 4(a), a network composed of uniformly distributed and randomly arranged AgNWs was embedded in PDMS to form a composite structure. Although the overlapping points in the AgNW network were covered by PDMS, their contact areas did not significantly increase. Thus, the pristine AgNW–PDMS composite electrodes showed high \( R_s \) values (figure 3(d)). However, most of the overlapping points in the AgNW network appeared to be in the fused state after 5 V sintering voltage treatment for 2 min, which has the advantages of simple treatment and short process time, and the local joule heat generated at the intersection effectively reduced the contact resistance of the network structure (see figure 4(b)). However, when 5.5 V sintering voltage was continuously applied to both ends of the AgNW–PDMS composite electrode, the excessive joule heat caused fusion and separation of the AgNWs at part of the overlapping points in the network, as shown by the dashed circle in figure 4(c). This indicates that applying excessive sintering voltage to both ends of the AgNW–PDMS composite electrode will lead to a sudden increase of the \( R_s \) value.

In addition to eliminating the contact resistance in the AgNW network, the joule heat generated during the electro-sintering process on the AgNW–PDMS composite electrode can be used as an electrical transparent heater. To investigate the electric heating performance of the AgNW–PDMS composite structure, ITO with the same size (25 mm in length and width) and \( R_s = 8 \) ohm sq\(^{-1}\) was used as a comparison, and the distance between the two ends of the electrode to the applied voltage was also 10 mm. Even the AgNW–PDMS composite electrode with low deposition density of 240 mg m\(^{-2}\) achieved rapid thermal feedback at low voltage (figure 5(b)). Moreover, it took more than 400 s for the temperature of the ITO semiconductor heater to fall to room temperature after cutting off the voltage (figure 5(a)). By contrast, owing to the advantage of the higher metal thermal conductivity than those of semiconductor materials, the AgNW–PDMS composite electrode rapidly reached an approximate electric heating saturation temperature in 10 to 20 s, and only approximately
90 s cooling time was required (see figure 5(b) and (c)). Moreover, the temperature of the electric heating did not greatly change with time after reaching saturation. Lower electric heating voltage was required for higher deposition density of the AgNW–PDMS composite electrode, which is suitable for use under special conditions, such as vehicle voltage and button-battery-powered devices. Owing to the ultra-thin and foldable AgNW–PDMS composite structure, the composites have the advantages of being closely attached to the contactor shape (see video 1 in the supplementary material), excellent biocompatibility and tensile properties, and they are expected to be suitable as the fundamental electrodes of future electronic skin or devices at human joints (see video 2 in the supplementary material). In view of the gas permeability and thermal stability of PDMS materials, the AgNW–PDMS composite film can be made into an electric heater at a human joint to eliminate the pain caused by arthritis with a button-battery alone, and electric heaters can be used at many small joints, such as finger joints, without affecting the normal activities of the hands.

Environmental and mechanical stability are important factors for using the AgNW–PDMS composite structures as electrodes in optoelectronic devices. Using the AgNW–PDMS composite structure as an electrode for skin electronics or as a faying electrode to construct human signal detection devices has high requirements on the stability. Therefore, the AgNW–PDMS composite structure was compared with the AgNW network deposited on a glass substrate in an aging test under high temperature and high humidity (∼85 °C and 85% relative humidity (RH) conditions) (figure 6(a)). The $R_s$ value of the AgNW network deposited on the glass substrate significantly increased after 2 h in an aging environment and increased by 32.7 ohm sq$^{-1}$ after 24 h. In contrast, owing to the hydrophobicity (see video 3 in the supplementary material) and good heating stability of the AgNW–PDMS composite, $R_s$ remained stable and only increased by 2.4 ohm sq$^{-1}$ after aging for 24 h. The composite structure showed the characteristics of temperature and humidity resistance, so it can be used as a transparent bonding electrode for skin. For example, it can be used as an electrode to construct a chip for recording the electrocardiogram without being affected by the patient’s sweat. In addition, the AgNWs coated by PDMS showed higher voltage resistance than those exposed on a glass substrate. The AgNW–PDMS composite structure showed the best sintering effect at 5 V (figure 6(b)), that is, the contact resistance had the least influence. However, for the AgNW network deposited on a glass substrate, the phenomenon of fusion separation occurred under this voltage, and then the interelectrode resistance sharply increased (figure 6(b)).

The mechanical stability, mainly focusing on the recovery ability and electrical properties of the composite structure after complete folding, is also important. It is now not satisfactory for transparent electrodes to only have stability after bending to a small radius of curvature, and maintenance of the electrical performance under
complete folding or even multiple folding in half, which is beneficial for expanding the applications of flexible transparent electrodes, is also important. Therefore, the AgNW–PDMS composite electrode (width and length of 25 mm) with deposition density of 320 mg m$^{-2}$ was folded in half under pressure of mechanical springs different number of times, as shown in figure 6(c), and the variation of $R_s$ was measured after each restoration (see figure 6(d)). The $R_s$ value of the AgNW–PDMS composite electrode changed little after completely folding in half once and twice, and it only increased by 1.1 and 2.9 ohm sq$^{-1}$ when it was folded in half three and four times, respectively. The combination of ultra-flexibility and robustness of the PDMS material and AgNWs enables this composite structure without an attached substrate to be able to recover after folding in half multiple times (see video 4 in the supplementary material). The mechanical stability during extreme stretching and bending is important for multi-functional applications of the AgNW-network electrodes.

In order to preliminarily explore the possible applications of the flexible AgNW–PDMS composite electrode in electrochemical devices, we tested the cyclic voltammetry (CV) performance of the AgNW-network electrodes. Figure 7 shows the CV curves of AgNW-network electrodes at a scan rate of 10 mV s$^{-1}$ at potential window of 0–0.6 V in 2 M NaOH aqueous electrolyte. The three-electrode system consists of the obtained AgNW-network electrode, a platinum foil, and Hg/HgO. They serve as working electrodes, counter electrode and reference electrode, respectively. The CV curves of the AgNW network and AgNW-PDMS composite electrode with deposition density of 400 mg m$^{-2}$ both exhibit a pair of redox peaks, which can be attributed to the reversible redox couple Ag/Ag$^+$ as follows:

$$2\text{Ag} + 2\text{OH}^- \leftrightarrow \text{Ag}_2\text{O} + \text{H}_2\text{O} + 2\text{e}^-$$ (1)

This can offer a potential application of AgNW-based networks for electrochemical devices in self-powered flexible electronic apparatus.

4. Conclusions

A AgNW-network-embedded PDMS composite structure has been prepared by a simple process. The AgNW-network electrode showed transmittance of 84% and 73% at 550 nm with $R_s$ values of $\sim$82.9 and 12.6 ohm sq$^{-1}$, respectively. This ultra-thin and substrate-free transparent film is suitable as the electrode of skin electronics and wearable devices. The $R_s$ value of the randomly arranged and flexible AgNW-based composite can be effectively reduced by electro-sintering treatment. Electro-sintering can weld the overlapping AgNWs, and the $R_s$ values of AgNW–PDMS composite structures with deposition concentrations of 240 and 320 mg m$^{-2}$ greatly decreased by 285.7 and 23 ohm sq$^{-1}$, respectively. It was found that the joule heat generated in the AgNWs, a material with high thermal conductivity, leads to much shorter heating and cooling times than metal oxide materials in the process of electric injection. The small electric heating voltage is suitable for vehicle voltage and button batteries. By embedding the AgNW network in PDMS, the influence of a high-temperature and high-RH aging environment can be greatly reduced (2.4 ohm sq$^{-1}$ increase after 24 h aging), and the good toughness and non-substrate binding make the AgNW–PDMS composite show excellent recovery ability and electrical stability after folding multiple times (2.9 ohm sq$^{-1}$ increase after folding four times). This flexibility will allow the AgNW–PDMS electrode to be efficiently attached to the skin at bones or joints, making it suitable for future skin electronics or wearable devices.
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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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