Facile fabrication of large-scale silver nanowire transparent conductive films by screen printing

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Abstract
Silver nanowire transparent conductive films (AgNW TCFs) were facilely prepared by screen printing conductive ink on a polyethylene terephthalate (PET) substrate, and the effects of ink compositions and oily stencil on the optoelectrical properties of AgNW TCFs were investigated in detail. 7.3 mg ml\(^{-1}\) hydroxypropyl methylcellulose (HPMC), 4.12 mg ml\(^{-1}\) AgNWs and 98T oily stencil allow the preparation of large-scale AgNW TCFs with high transmittance, low square resistance and high uniformity. The resultant screen printed AgNW TCFs possesses a sheet resistance as low as 13.0 ± 0.6 Ω sq\(^{-1}\), a transmittance of about 95.3% at 550 nm wavelength (deducting the background) and a haze of 3.86 (deducting the background), and can achieve a surface root mean square roughness of 3.33 nm, a film size of 15 \times 20 cm\(^2\) and personalized pattern by means of the screen printing process. The transparent film heater (TFH) constructed by AgNW TCFs can rise to a usable temperature of 55 °C at a low voltage of 4 V within 80 s. This process provides a simple strategy for fabricating uniform, patterned and large size AgNW TCFs for various devices.

1. Introduction

Recently, metal nanowires transparent conductive films (TCFs) have attracted more and more attention due to many promising applications such as film heaters, solar cell electrodes, electromagnetic shielding devices [1–3]. The conductive layer of TCFs consists of an interlaced network of metal nanowires, and it retains a large number of voids so that the films are conductive and transparent at the same time [4–7]. The metal nanowires based TCFs are suitable for some special application scenarios such as window defogging, organic light-emitting diode (OLED) and electroluminescence devices [8, 9]. Metal nanowires based TCFs possess better electrical conductivity due to the excellent electrical conductivity of metal nanowires. Therefore, metal nanowires are ideal materials to replace indium tin oxide (ITO) [10, 11], carbon nanotubes [12], graphene [13, 14] and conductive polymers [15] to fabricate TCFs. Among metal nanowires, silver nanowires (AgNW) is the most promising candidate owing to excellent conductivity, relatively low cost and convenient fabrication [16–24].

Nevertheless, some defects restrict the commercial application of AgNW TCFs, including difficulty in large-scale production, personalized pattern and precision printing [25, 26]. Currently, ultrasonic spray coating [27–30], spin coating [21], slot coating [31–33] and screen printing [7, 34–44] have been used for the large-scale production of high-quality TCFs. Wherein, ultrasonic spraying as an efficient method to produce large-sized TCFs, while it has to face with defects such as high surface roughness and poor uniformity. Spin coating can achieve high uniformity, but it is difficult to get precisely pattern conductive electrodes. Slot coating is a developing printing process of TCFs, yet it is limited due to low efficiency and high cost. By contrast, screen printing as a traditional printing process has been widely used in fine electrode circuit printing because of its low...
cost and patterned electrodes, which combines uniformity and low surface roughness with excellent optoelectronic properties \[31\].

It is worth noting that AgNWs based conductive ink is a prerequisite for producing high-performance AgNWs network, and a suitable viscosity is equally critical for screen printing \[34, 45\]. Traditionally commonly used organic polymer thickeners are toxic, such as trimethylolpropane triacrylate (TMPTA), tripropylene glycol diacrylate (TPGDA) \[46\], polyvinylidene fluoride (PVDF) \[47\] and dimethylformamide (DMF) \[48\], and many post-processing steps are required, resulting in high cost and environmental pollution. Therefore, a new thickener with high transmittance should be developed to disperse AgNWs and increase the viscosity. High molecular weight hydroxypropyl methylcellulose (HPMC) possesses excellent transmittance and effectively increases the viscosity of the conductive ink. More importantly, AgNWs have good dispersibility in HPMC solution, and HPMC wrapped around silver nanowires as a certain protective role without negative effect on the conductivity of silver nanowires. The stability and water solubility of HPMC facilitate the preparation of conductive inks. Therefore, it may be feasible to disperse AgNWs with HPMC solution instead of traditional thickener.

In this work, we report a simple strategy for fabricating uniform, patterned and large-scale AgNW TCFs by screen printing process. The conductive ink for screen printing is formulated by dispersing AgNWs with HPMC solution and dispersing agent FSO-100. The conductive ink was screen printed on a polyethylene terephthalate (PET) substrate to fabricate large-scale and customized patterns AgNWs TCFs with uniform optoelectronic properties, without subsequent etching processing. The transparent film heater constructed by AgNWs TCFs displayed high heating performance at low input voltage and high heating cycles stability.

2. Experimental

2.1. Materials and fabrication of AgNWs TCFs

AgNW dispersion with an average diameter of 25 nm and an average length of 20 μm was purchased from Zhejiang Kechuang Advanced Materials Technology Co., Ltd Hydroxypropyl methylcellulose (HPMC; VIS = 100000 mPa·s) was obtained from Ourchem Co., Ltd, and FSO-100 (0.1 mg·ml⁻¹) from DuPont Co., Ltd

In a typical experimental, 0.472 g HPMC was dispersed in 15 g 99.7% alcohol solution in a beaker, and then 25 g deionized water and 1 ml FSO-100 were added into the HPMC dispersion. The mixed solution was stirred vigorously until HPMC was fully dissolved. After at least 30 min, the HPMC solution was put into an ultrasonic cleaner until the bubbles disappeared. After the above operation, 28 ml AgNW dispersion with a concentration of 10 mg·ml⁻¹ was well mixed with HPMC solution, after stirring for 10 min, AgNW conductive ink for screen printing was prepared.

AgNW TCFs were fabricated by a screen printing process of AgNW conductive ink on 15 cm × 20 cm PET substrates. The AgNWs conductive ink was printed at the speed of 3 ~6 cm·s⁻¹ with an off-screen spacing of 5 mm. Finally, the fabricated AgNW TCFs were treated at 50 °C for 10 min to form stable electrode layer. Heating films (5 cm × 5 cm) was constructed by taking any appropriate size on TCFs and printing two silver electrodes (5 cm × 0.5 cm) on both ends.
2.2. Characterization
Scanning electron microscopy (SEM) images of AgNW TCFs on PET substrates were obtained using an SU8010 high resolution microscope from Hitachi with an accelerating voltage of 3 kV. Four-probe surface resistivity meter (RTS-9, Guangzhou Four-point Probe Technology) was applied to measure the sheet resistance of the composite flexible TCFs, and the transmittance was measured by a TU-1810 UV–vis spectrophotometer from Beijing Purkinje General Instrument Co., Ltd Surface roughness was collected by an atomic force microscope (AFM) (Dimension Icon, Bruker, Germany) using a tapping mode. Viscosity was performed via BROOKFIELD VISCOMETER DV-II+ Pro. Voltage, current and power were carried out by PRECISE S100 Source Meter and the real-time temperature was recorded through CENTER 304/309 (DataLogger). The DC voltage supply and current measurement were provided by a Precise S100 source meter. The heating temperature of the AgNW TCF transparent heater was monitored using a CENTER 309 temperature detector adhered to the middle point of the bottom of the heater and continuously monitored using data collection software. The infrared images were recorded using a DL 700E infrared thermal imager.

3. Results and discussion
3.1. Construction of AgNW TCFs by screen printing
The screen printing process of AgNW TCFs on PET substrates is shown in figure 1. To obtain conductive ink with suitable viscosity, the HPMC is firstly dispersed into absolute ethanol to avoid the agglomeration of HPMC and obtain a viscous HPMC solution, and the obtained HPMC solution is used as the ink base to disperse the silver nanowires as well as play a certain protective role for silver nanowires. The silver nanowires are further added and dispersed into HPMC solution without breaking under high-speed stirring to prepare conductive ink. The formulated conductive ink is screen printed on a PET substrate, and AgNW TCFs can be prepared after heating and drying. A transparent film heater can be constructed by printing silver paste electrodes on both ends of the obtained transparent conductive film.

In the screen printing process, there is a gap between the stencil and the substrate. During the movement of the squeegee, the viscosity of the ink will be reduced due to the shear force between the squeegee and the stencil. The ink flows through the mesh on the screen onto the PET substrate to form a uniform thin film, and AgNW TCFs are achieved after heating and drying.

3.2. Effect of conductive ink on photoelectric properties of AgNW TCFs
The screen printing process depends on the viscosity and leveling performance of the conductive ink, which are directly related to the optoelectronic properties and uniformity of the TCFs. The conductive ink mainly consists of HPMC solution and AgNWs dispersion, and the HPMC solution disperses AgNWs and increases the viscosity...
of conductive ink. The concentrations of HPMC solution and AgNWs dispersion have an important role on the photoelectric properties of AgNWs TCFs.

Figure 2 shows the variation of the viscosity of conductive ink and the photoelectric properties of TCFs with different HPMC concentrations. It is seen from figure 2(a) that when the HPMC concentration increases from 5 to 11 mg·ml⁻¹, the viscosity of the conductive ink increases from 515.9 to 7486 mPa·s with almost exponential growth, and the transmittance of the AgNWs TCFs slightly increases from 97.5% to 99.4% (deduct the background and measured at 550 nm band). It can be observed from figure 2(b) that the sheet resistance of the AgNWs TCFs firstly decreases and then increases with the increase of HPMC concentration. When the HPMC concentration is 7.3 mg·ml⁻¹, the sheet resistance reaches a minimum of 55.4 Ω sq⁻¹.

The sheet resistance of 12 points on the film is collected, and the variance of the sheet resistance is calculated by the following variance formula:

\[ s^2 = \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n} \]  

wherein, \( \bar{x} \) and \( n \) represents the average number of measured sheet resistances and the number of measured sheet resistances, respectively. The calculation result is shown in the figure 2(b). It is noted that the variance of sheet resistance decreases firstly, and then increases with the increase of HPMC concentration. A minimum variance of sheet resistance is 2.85 at a concentration of 6.9 mg·ml⁻¹, indicating that too low or too high HPMC
concentration is detrimental to the uniformity of the TCFs. Figures 2(c)–(e) show the SEM images of AgNW TCFs with high HPMC concentration. There are some aggregation and evacuation of the silver nanowire network. In comparison, the appropriate concentration of HPMC is 6.9 mg·ml$^{-1}$ for AgNW TCFs.

**Figure 4.** (a) Sheet resistance, variance of sheet resistance and (b) transmittance spectra of AgNW TCFs with different screen mesh numbers; sheet resistance of AgNW TCFs with different printing speeds (c) and off-grid spacings (d).

**Figure 5.** SEM images of AgNW TCFs screen printed with mesh numbers of 138T (a), 118T (b), 98T (c) and 79T (d).
The effect of AgNWs concentration in conductive inks on the photoelectric properties of the films were further investigated. Figure 3 shows SEM images and sheet resistance of screen printed AgNW TCFs with different AgNWs concentrations. It is seen that with the increase of AgNWs concentration, the AgNWs in the conductive network display a change from sparse to dense, while the sheet resistance and the variance of sheet resistance of AgNW TCFs decreases rapidly. The rate of reduction of square resistance significantly slows down at the AgNWs concentration of $2 \sim 3$ mg ml$^{-1}$, which is consistent with penetration theory. The sheet resistance variance decreases gradually at first, and remains basically unchanged at 0.3 ± 0.1 when the AgNWs concentration reaches 3.6 mg ml$^{-1}$. The transmittance of AgNW TCFs is gradually reduced with the increase of AgNWs concentration. As a result, the AgNWs concentration of 3.6 mg ml$^{-1}$ is suitable for conductive ink.

### 3.3. Effect of screen printing on photoelectric properties of AgNW TCFs

Commonly, the types and meshes of screen printing stencils, printing speed and the distance between the substrate and stencils will affect the microstructure and photoelectric properties of screen printed film. Figure 4 shows the photoelectric properties of AgNW TCFs with different screen mesh numbers, printing speeds and off-grid spacings. It is noted that the mesh number of stencil has a significant impact on the photoelectric properties of AgNW TCFs. With the increase of mesh number, the sheet resistance of AgNW TCFs increases slowly, while the transmittance raises correspondingly. When the mesh number is more than 98T, the sheet resistance increases exponentially, and the transmittance is further improved. The sheet resistance of the composite films increases from 6.2 to 185.4 $\Omega$ sq$^{-1}$, and the transmittance has also been greatly improved from 86.3% to 99%, with the mesh number increasing from 47T to 165T. It can be also seen from the variance of sheet resistance that when the mesh number exceeds 98T, the variance of sheet resistance rises sharply. In contrast, it is observed that the sheet resistance and the variance of sheet resistance change little with the increase of printing speed and off-screen distance, which indicates that the printing speed and off-screen distance have little effect on the photoelectric properties of AgNW TCFs.

Figure 5 show the SEM images of AgNW TCFs with different screen mesh numbers. It is obviously seen that there are more and more AgNWs in the conductive network with the stacking of AgNWs, which indicates that AgNW TCFs has high conductivity, and while the transmittance will be deteriorated. By comparison, the morphology of AgNWs in the conductive network is clearer, and the distribution of AgNWs is more uniform. In fact, at the mesh number of 98T, the average sheet resistance of AgNW TCFs is 13.0 $\Omega$ sq$^{-1}$ with a sheet resistance variance of 0.3, and the transmittance is 95.3% with a haze of 3.86, displaying high photoelectric properties. It confirms that the mesh number of 98T is appropriate for screen printing of conductive ink.

Figure 6 shows the SEM, AFM and pattern of AgNW TCFs screen printed at the mesh number of 98T. It is observed that AgNWs are stacked and connected to each other to ensure the conductivity of the network (figure 6(a)), and the voids in the network retain the transparency of AgNW TCFs. The HPMC coats the AgNWs, and also fills the voids of the AgNW network. The root mean square roughness of AgNW TCFs can reach...
3.33 nm (figure 6(b)), indicating low surface roughness. The obtained AgNW TCFs has excellent electrical conductivity (figure 6(c)), and different screen plates can be customized through the screen printing process to meet the patterning requirements of TCFs (figures 6(d)−(f)).

3.4. Heating performance of AgNW TCFs based transparent film heater

As shown in figure 1, AgNW TCFs based transparent film heater can be fabricated by printing two silver paste electrodes on a 5 cm × 5 cm square area. The resistance between the two electrodes is 8.6 ± 0.5 Ω, and the resistance of the entire silver paste electrode is 1.9 ± 0.2 Ω. Therefore, the transparent film heater is allowed to heat up rapidly and uniformly at a very low voltage.

Figure 7(a) shows the temperature-time (T-t) curves of the heating film under voltages of 3, 4, 5 and 6 V. The transparent film heater can reach the maximum temperature within 80 s when the applied voltage is higher than 3 V. The higher voltage applied across the electrodes, the heating rate becomes faster and the corresponding maximum temperature is higher. It is worth mentioning that the temperature of the heater can reach 50 ∼55 °C at only 4 V, which is the appropriate temperature for defogging. And the uniformity of the heater can be shown from the infrared thermal image in figure 7(d), in which the difference between the highest and lowest temperature of the heating film is no more than 5 °C, indicating uniform heating performance.

A T-t step curve as shown in figure 7(b) was obtained by continuously increasing the voltage applied across the heater. The heater fails and has no conductivity after applying 8 V voltage for 28 s, and the maximum temperature reaches 117.5 °C. The reason for the failure of the heater is shown in SEM images before and after
the failure of the heater (figures 7(e) and (f)). It can be seen that the AgNWs in the heating film are fused at high temperature, causing the conductive network to be destroyed. The heating stability and heating performance of the transparent film heater are equally important, figure 7(c) shows the results of 26 cycles of heating at 4 V voltage. Each cycle includes voltage applied and no voltage applied for 60 s. The maximum temperature of each cycle is around 55°C, and the resistance of the transparent film heater before and after the cycle is measured within the range of 8.6 ± 0.5 Ω. Furthermore, the optoelectronic properties and heating performance of transparent film heater have also no change after 60 days, showing great stability.

4. Conclusions

AgNWs conductive ink was screen printed to prepare silver nanowire transparent conductive films (AgNW TCFs). The HPMC solution can well disperse AgNWs and provide suitable viscosity and rheology for conductive ink in screen printing process, and the concentrations of HPMC solution and silver nanowires and the mesh number of the stencil play important roles on the morphology and photoelectric properties of AgNW TCFs. Based on the fabricated AgNW TCFs, the transparent film heater can heat up rapidly at low voltage and reach usable heating temperature with excellent heating uniformity and stability.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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