High-Conductivity Screen-Printable Silver Nanowire Ink for Optically Transparent Flexible Radio Frequency Electronics

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Received xxxxxx
Accepted for publication xxxxxx
Published xxxxxx

Abstract

Optically transparent conductors have paved the way in various optoelectronic and radio frequency (RF) devices where high electrical conductivity and optical transparency with mechanical flexibility, as well as large area fabrication are deemed necessary. Printing techniques are viable for fabricating large-area devices with high mechanical flexibilities. However, the preparation of suitable inks and printing recipes is essential to achieve a high electrical conductivity and transparency. In this study, the best tradeoff between conductivity and optical transmittance was achieved through silver (Ag) nanowires (NWs)-based ink formulation with tuned Ag NW loading, solvent compositions and polymer weight percentages. The ink was deposited through screen-printing, which enabled a large-area and high-resolution patterning of the AgNWs. The washing time of the post-printed films exhibited a decisive effect on the initial conductivity, which was further improved through photonic sintering. During the photonic sintering, the voltages, pulse lengths (µs) and fire rates (Hz) were optimized to obtain the best conductivity of the printed films. Maximum optical transparencies of 78 % and 83 % were achieved for the conductivities of ~5.88 × 10⁷ and ~6.25 × 10⁷ S/m, respectively. As a proof of concept, a fully printed optically transparent antenna was realized that could operate in a wide frequency band suitable for high-data-rate wireless communication.

Keywords: transparent electronics, silver nanowires, conductive, flexibility, screen-printing, photonic sintering, antenna

1. Introduction

Recently, optically transparent and mechanically flexible conductive films have shown their utility for several emerging devices such as smart windows, touch screens, light emitting displays, solar cells, sensors, radio-frequency (RF) electronics, etc. [1-8]. Particularly, transparent RF electronics would be beneficial in many smart city applications without interfering aesthetically or physically with the surrounding environment, and thus, can be employed for various emerging
RF applications, such as reconfigurable intelligent surfaces [9-10], frequency selective surfaces [11-12], electromagnetic absorbers [13], RF shielding [14], antenna [15-16], etc. However, a high conductivity while balancing optical transparency is deemed necessary for these applications. In addition, for millimeter wave (mm-wave) RF operations, conductor losses are expected to increase; thus, a higher conductivity is always a prerequisite for such designs. To achieve a high optical transparency and conductivity simultaneously, various emerging materials have been explored in the last decade, including conductive polymers [17], carbon materials [18-19], and transparent conductive oxides [20-21]. For most of these materials, transparency is achieved by thin-film deposition using physical vapor deposition (PVD) or mesh formation with patterning solid metals. Particularly, the metal mesh formation is achieved by depositing a metal such as Ag, Au, Cu, and so on, followed by etching of the deposited metal on the substrate for patterning. However, the mesh fabrication techniques typically require complicated processes and involve excessive wastage of the raw materials. Furthermore, they require expensive masks and nanolithography for patterning. In contrast, metal nanowires (NWs), such as AgNWs, are promising owing to their inherent optical transparency and solution processability [22-24]. In addition, printing techniques allow a highly scalable manufacturing of transparent AgNWs films through a completely digitised and simplified process with extremely low costs [25-27]. However, to date, only a handful of studies have been reported on AgNW-based inks as listed in Table I, which also presents the respective conductivities and transparencies of these reported inks. There is always a tradeoff in achieving a higher conductivity and optical transparency simultaneously. As shown in Table I, for some inks with high conductivities (>10⁴ S/m) or low sheet resistances (<1 Ω sq⁻¹), the corresponding transparencies are poor (completely opaque) [28-30]. In contrast, some other inks with high optical transparencies (more than 50 %) exhibit low conductivities (about 10⁴ S/m) [31-34]. Previously, our group developed a screen-printable AgNW ink using a special procedure of welding the NWs [35]. These welded AgNWs demonstrated the best reported tradeoff between conductivity (5.5 × 10⁴ S/m) and transparency (~75 %). Although balancing this tradeoff is a considerable challenge, it is an essential requirement for many practical applications, where even better conductivities and transparencies are needed. For example, in RF devices such as transparent antenna, a higher conductivity is deemed important while balancing the transparency to more than 80 %.

Driven by the high demand of RF devices with high conductivities and transparencies, we optimized two different ink formulations by varying the Ag NW loading, solvent compositions and polymer weights (%) in the inks. It was observed that the washing time for such inks influenced the initial conductivity of the printed films, which was further improved via photonic sintering. During the photonic sintering, the voltages, pulse lengths (μs) and fire rates (Hz) were optimized to obtain the best conductivity of the printed films. By optimizing the ink composition with photonic sintering, we obtained the maximum transparencies of 78 % and 83 % for the conductivities of ~5.88 × 10⁴ and ~6.25 × 10⁵ S/m, respectively. In addition, the printed films were tested under extreme bending conditions. As a proof of concept, we fabricated a highly transparent antenna through fully screen-printing process, which operates in a wide frequency band.

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<th>Sheet resistance (Ω sq⁻¹)</th>
<th>Optical transparency (%)</th>
<th>Ref #</th>
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2. Experimental Details

2.1 Chemicals

Silver nitrate (AgNO₃, Sigma-Aldrich, ACS reagent, ≥99 %), ethylene glycol (EG, C₆H₁₂O₆, Sigma-Aldrich, spectroscopic grade, ≥99 %), iron (III) chloride hexahydrate (FeCl₃·6H₂O, Sigma-Aldrich, reagent grade, ≥98 %), polyvinyl pyrrolidone (PVP; K30, M_w = 55,000; K90, M_w = 360,000; Sigma-Aldrich), 1, 2-propanediol (C₃H₆O₂, Sigma-Aldrich, puriss, ACS reagent, ≥99.5 %) and ethanol absolute (C₂H₅OH, VWR chemicals, >99.7 %) were used. PVP K-120 (M_w = 2,000,000) was obtained from Ashland. These chemicals were used as received without further purification.

2.2. Preparation of AgNWs

As shown in Fig. 1, first, we prepared a base solution by adding 0.8 g of PVP K30 (M_w = 55,000) and 0.8 g PVP K90 (M_w = 360,000) into 200 mL of ethylene glycol, and stirred this mixed solution at 600 rpm until the PVP was totally dissolved. Then, 2.1 g of AgNO₃ was added to this base solution. In the second step, a seed solution was prepared by adding 0.0162 g of FeCl₃ (0.06 M) into 100 mL of ethylene glycol and stirred at 600 rpm until the components were totally dissolved. Next, a 250 mL flask was placed into an oil bath heated to 120 °C until the temperature was stabilised, as shown in Fig. 1(a). A precursor solution was prepared by adding 30 g of the seed solution to the base solution and stirred for 2 min to form the precursor solution, which was subsequently transferred to the heated flask and capped (Fig. 1(b)). The reaction proceeded for almost 5 h, and the precursor solution turned greyish in color. During this reaction, first, nucleation occurs (Fig. 1(c)), followed by the growth of NWs (Fig. 1(d)). Here, ethylene glycol acts as both a solvent and a reducing agent, AgNO₃ provides the Ag ions, and PVP acts as a capping agent, which directs the anisotropic assembly of the Ag atoms (nuclei) into NWs. In addition, FeCl₃ acts as an etchant that enables the polyol reduction to proceed at a slower rate, thereby facilitating a uniform growth of the AgNWs [36]. After 5 h of the reaction, the heating was stopped, and the flask was cooled to room temperature. Then, the cooled solution was poured into a 1 L beaker and acetone, with a volume twice that of the cooled solution, was added to the solution under continuous stirring for 1 min. Following this procedure, the AgNWs precipitated at the bottom of the beaker (Fig. 1(e)). Subsequently, the supernatant was discarded and 50 mL of de-ionized (DI) water was added to the precipitate to redisperse the AgNWs under stirring at 500 rpm (Fig. 1(f)). Finally, the acetone addition and re-dispersion with DI water were repeated two to three times to remove the excess polymers. The wet precipitate of AgNWs after washing was used for the preparing the ink as explain in the next section.

![Figure 1. Schematic of the synthesis of AgNWs.](image)

2.3. Ink formulation and Screen printing of the AgNWs

The inks were formulated by mixing the washed AgNWs with a solution containing PVP (K-120), propandiol and ethanol. The weight percentage of the AgNWs and PVP as well as the solvent composition of propandiol and ethanol were optimized with respect to transmittance and sheet-resistance, as mentioned in the text while discussion.

The large-area AgNWs patterns were printed on an ethanol-cleaned polyethylene naphthalate (PEN) substrate. A screen stencil with a mesh count of 325 µm, a wire diameter of 20 µm, and an emulsion thickness of 10 µm, was used for the printing. The PEN substrate was attached on a plate, and the as-formulated ink was placed in front of a squeegee. A printing speed of 220 mm/s was used to spread the ink and transfer the pattern on the targeted substrate. After printing, the sample was dried in an oven at 80 °C for 5 min. The over-dried samples were then immersed into water for 30–60 min to remove the polymer (PVP K120). After the polymer removal, the samples were dried again in the oven at 80 °C for 5 min.

2.4. Photonic Sintering

The printed samples, after washing and drying, were subjected to photonic sintering (Novacentrix Pulse Forge 1300 system). The printed sample was placed at a constant distance of 5 mm from the flash light throughout the sintering process. Several settings of the photonic sintering system, such as voltage, pulse length (µs) and fire rate (Hz), were optimized as mentioned in the text.

2.5. Fabrication of Fully Printed Transparent Antenna

The computer simulation technology microwave studio was utilized to simulate a circular monopole antenna. A three-dimensional (3D) model, containing two conductor layers (one for antenna and another for ground plane) and a dielectric layer (PEN substrate), was constructed. The dielectric substrate had a dielectric constant of 2.7 and a dielectric loss
tangent of 0.0047 at 1 GHz. The substrate thickness was 125 μm, and the conductivity and thickness of the AgNW film were considered to be $6 \times 10^6$ S m$^{-1}$ and 0.1 μm, respectively. A waveport of 50 Ω was modeled as the source.

2.6. Characterizations

The morphologies and crystalline structures of the AgNWs were characterized by scanning electron microscopy (SEM; ZEISS Merlin) and X-ray diffraction (XRD; Bruker D8 ADVANCE), respectively. The rheological behaviour of the as-formulated AgNW inks were tested using an AR1500 rheometer (TA Instruments). Thermal gravimetric analysis (TGA) of the inks was performed using a TGA-TA Discovery 5500 instrument. The transparency was evaluated using ultraviolet-visible spectroscopy (UV-Vis; UV-Vis-NIR-Lambda 950). The sheet resistance of the AgNW film was measured using an M-3 mini type four-probe resistance tester (Suzhou Jingge Electronic Co., Ltd.). The thickness of the AgNW film was measured using a surface profiler (Dektak profilometer). RF measurements were performed using an Aglient N5225A Vector Network Analyzer. The 5-parameters of the antennas were acquired from 4 to 7.5 GHz after RF calibration, which was conducted based on the short-open-load method, to eliminate systematic errors from the measurement setup. The antenna gain and its radiation pattern were obtained in a near-field anechoic chamber (Satimo Sturlab). Before the measurements, the chamber was calibrated for the frequency of interest.

3. Results and Discussion

The sizes of the prepared AgNWs were confirmed from their corresponding SEM images. Fig. 2 displays the low-resolution and high-resolution SEM images of the NWs synthesized through a 5-h reaction and washed with DI water and acetone. From the SEM images, it could be confirmed that the NWs had an average diameter of 45 ± 5 nm and a length of 25 ± 5 μm. The typical XRD pattern of the AgNWs exhibited diffraction peaks indexed to the face-centered cubic Ag without any impurities [37]. It was observed that the intensity of the Ag (111) peak was much higher than that of the Ag (200) peak, and this higher intensity Ag (111) peak corresponded to the high aspect ratio of the AgNWs.

After confirming the morphology and purity, the as-prepared AgNWs were used for preparing the inks. To achieve the best combination of transmittance and sheet resistance, several ink formulations were developed while maintaining a decent viscosity for screen printing. Two different ink formulations (Ink A and Ink B) were optimized by varying the AgNW loading, solvents (propanediol and ethanol) and polymer weight % (PVP K120) in the inks. Initially, an ink vehicle was prepared by adding 3 wt% PVP K120 (M_w = 2,000,000) into 54 wt% of propanediol and 40 wt% ethanol (1.35:1 weight %), and the resulting solution was stirred at 600 rpm until the added components were totally dissolved. Next, the AgNW precipitate (~3 wt%) was added to the ink vehicle and then agitated until totally dispersed. The as-resultant ink was termed as Ink A. The ink was spread with a speed of 220 mm/s on the PEN substrate to prepare the printed films, which were found to be dense (as shown in Fig. S1); thus, a sheet resistance of 2–2.5 Ω/sq at 67 % transparency was observed after washing the film with DI water. As the transparency was very low with the as-prepared Ink A, it required further optimizations; that is, the ink was further diluted using propanediol and ethanol in the ratio of 2:1. For example, 80.12 wt% (2 g) of Ink A was diluted with 13.22 wt% (0.33 g) of propanediol and 6.65 wt% (0.166 g) of ethanol (Ink A1), and in the second formulation, 72.72 wt% (2 g) of Ink A was diluted with 18.18 wt% (0.5 g) of propanediol and 9.08 wt% (0.25 g) of ethanol (Ink A2). However, further dilution beyond Ink A2 leads to unreliable printing as the ink splashes on the screen of the printer. It should be noted that with such dilution, the weight % of AgNW is also decreased. In addition, it is very important to have a balance between the solvent composition and the polymer weight % where both can effectively contribute to the decent viscosity of the ink. Based on the observation with Ink A, another ink formulation (Ink B) was prepared. The ink vehicle was prepared by adding a comparatively low weight % of PVP, such as 2.5 wt% PVP K120, into 86 wt% propanediol and 10 wt% ethanol (8.6:1 weight %), and then the solution was stirred at 600 rpm until the components were totally dissolved. Then, 1.2 wt% of the AgNW precipitate was added to the ink vehicle and agitated until the precipitate was totally dispersed. It should be noted that the mixed solvent of propanediol and ethanol was selected.
owing to its high viscosity and low surface tension. In addition, PVP was used as an organic binder, a dispersing agent as well as a rheological modifier.

Because the inks were diluted with the mixed solvents, their thixotropic behaviour was evaluated before the printing. To confirm the thixotropic behaviour, the viscosity of the resultant inks were measured using a rheometer at a shear rate from 0.1 to 1000 s⁻¹, and the corresponding curves are displayed in Fig. 3. We clearly observed a shear thinning behavior of the as-formulated AgNW inks. The viscosity of Ink A decreased from 2076 to 202 cP as the shear rate increased from 0.1 to 10 s⁻¹. Similarly, the diluted Ink A1 and A2 showed a decrease in the viscosity from 1476 to 198 and 1234 to 180 cP, respectively with a shear rate in the range from 0.1 to 10 s⁻¹. Due to the less weight % of PVP and AgNWs, Ink B showed further decrease in its initial viscosity (1100 cP at 0.1 s⁻¹) and also exhibited a typical shear thinning behaviour as the shear rate was increased (89 cP at 10 s⁻¹). This type of fluid behavior and decent viscosity are essential for a favorable printability.

Next, to confirm the loading of the AgNWs, the as-formulated inks were further characterized using TGA, as shown in Fig. 4. The TGA results confirmed a sharp decrease in the weight % loss in between 70 and 200 °C; this corresponds to the temperature range at which the ink solvents (propandiol and ethanol) evaporated for all the inks. The weight % loss in between 400 and 500 °C was mainly due to the thermal decomposition of the PVP K-120 polymer. The weight % beyond 600 °C corresponded to the solid loading of the AgNWs in the inks, as shown in Fig. 4 (b). The added weight % of the AgNWs in Ink A well matched that of the corresponding thermally decomposed Ag (2.97 wt%). The diluted inks Ink A1 and A2 exhibited final solid loadings of 2.26 and 2.06 wt% of the AgNWs, respectively. Ink B resulted in a solid loading of 1.2 wt% of the AgNWs. After the ink characterizations, these inks were used to print large-area patterns, whose sheet resistances and transmittances were investigated as well.

Patterns were screen-printed using both Ink A and Ink B by using a screen-printing system (AUREL screen-printer 900PA) at a speed of 220 mm/s. The printing, drying, washing and sintering processes are illustrated in Fig. 5(a)–(e). First, the desired patterns were transferred through a screen mask on the target PEN substrate (a). The printed films usually contained solvents that needed to be evaporated (b); thus, these printed films were dried in an oven at 80 °C for 5 min (c), followed by a washing step (d). After washing with DI water, the dried printed films were exposed to photonic sintering, which produced sintered films without affecting the substrate. The photonic sintering was mainly optimized by varying the voltage, pulse lengths and firing rates. It was observed that washing the printed films with DI water significantly influenced their initial sheet resistances. For example, the sheet resistance dropped from 4–5 Ω/sq to ~3 Ω/sq after washing the printed film for 5 to 60 min (Ink A). For Ink A, washing for even 60 min did not produce any adhesion problem. However, the corresponding sheet resistance did not decrease further when the washing time was increased beyond 60 min. Similarly, the sheet resistance dropped from 6–7 Ω/sq to ~4 Ω/sq while washing for 5 to 30 min when Ink B was used. Extending the washing time beyond 30 min affected the adhesion of the films printed using Ink B, mainly because of the small weight percentage of the polymer in Ink B. For Ink A and B, 60 and 30 min washing, respectively is the optimized time where excessive polymer washed away while maintaining the decent adhesion to the substrate. It should be noted that for effective photonic sintering, the AgNW films
should have minimum polymer content otherwise these polymers start burning during the sintering process which may have an adverse effect on the conductivity of the printed films.

The transparency of the films (at 550 nm) printed using Ink A and B are shown in Fig. 6 and their corresponding conductivities are summarized in Table S1 (supplementary data). The conductivities were calculated based on the average thicknesses of the films printed using Ink A and B, as shown in Fig. S2. A maximum conductivity of 5.05 × 10⁻⁶ S/m at 76% transmittance was observed for the film printed using Ink A1. Ink A2 resulted in a film with a maximum conductivity of 5.88 × 10⁻⁶ S/m at 78% transmittance, which was the optimized combination of transparency and conductivity obtained from among all the formulations of Ink A. The SEM images (Fig. 7 (a) and (b)) of the printed films showed welded NWs at their junctions (marked with yellow circle), which helped in increasing the number of conductive paths with photonic sintering. The best conductivity of ~6.25 × 10⁻⁶ S/m at 83% transmittance was obtained when the film was printed using Ink B. The SEM images shown in Fig. 7 (c)-(d) clearly show the welded NWs at the overlapping area, which aid in enhancing the conductivity of the printed films. The maximum conductivity with 83% transmittance was observed because of the small population of the AgNWs in the film, which was effectively sintered with the photonic light. In contrast, a thicker film, with more number of overlapping AgNWs, contained a small number of sintered hotspots responsible for the conductive path formation. This is primarily due to the penetration depth of photonic light. Thus, the conductivities are relatively lower for Ink A as compared to Ink B. It is important to note that the thinner films with a lesser number of overlapping AgNWs could be sintered effectively but that does not mean that further decreasing the AgNWs loading will help in enhancing the conductivity of printed films. There must be a balance between AgNWs loading and solvent composition for a decent viscosity needed for screen-printing. Thus, Ink B is the optimized combination of AgNWs loading and solvent composition to achieve the best conductivity while maintaining the transmittance greater than 80%.

The AgNW Ink B was further tested for the direct patterning of the AgNWs over a large area and high resolution. Fig. S3 presents digital photographs and microscopic images of the printed AgNW patterns with a transparency of 83% on the flexible PEN substrates with a dimension of 16 × 4 cm². The microscopic images shown in Fig. S3 (c)-(d) revealed the sharp and smooth edges of the screen-printed AgNW-based cross-over line and dot patterns. It should be noted that our printed AgNW films exhibited the lowest sheet resistance with a relatively high transmittance compared with those reported previously for AgNW-ink-based printed films [28-35], as summarized in Table 1.

![Figure 5](image1.png)

**Figure 5.** Fabrication steps of the proposed screen-printed transparent and conductive films.

![Figure 6](image2.png)

**Figure 6.** UV-Vis spectra of the as-printed AgNW-based films prepared using Ink A and B.

![Figure 7](image3.png)

**Figure 7.** Low- and high-resolution SEM images of the patterns printed using the AgNW ink on the PEN substrate after the photonic sintering: (a)-(b) film printed using Ink A2 and (c)-(d) film printed using Ink B.
After achieving the best tradeoff between conductivity and transmittance, the mechanical tolerance of the printed films was examined. To evaluate the tolerance of the printed films against bending, printed films with different transparencies were bended to a radius of 18 mm, as shown in Fig. 8a. Evidently, the printed AgNW-based films exhibited a robust tolerance to bending stresses up to 10,000 cycles. Because of the excellent electrical conductivity, high optical transparency, and robust mechanical stability, our AgNW-based films are ideal for application in various flexible electronic devices.

![Figure 8. Bending effect on the printed AgNW films over 10,000 bending cycles.](image)

To demonstrate the potential of the printed films for electronic applications, we applied the screen-printed flash-sintered AgNW-based films for antenna applications, where a high conductivity is critical for high performance. As a proof of concept, a fully printed circular monopole antenna was designed. The model and fabricated antenna prototype are displayed in Fig. 9 (a-c). First, a circular monopole with a radius of 4.5 mm was connected with a feeding line of 20.126 mm in length (L2) and 0.35 mm in width (Wf). Second, the ground plane was printed on the backside of the monopole antenna with a length of 20 mm (L1) and width of 40 mm. As demonstrated in Fig. 9 (d), the simulation results were well correlated with the measured results. However, slight variations were observed at higher frequencies (on right side) possibly due to the fabrication issues related to the placement of the SMA connector with Ag paste. The fabricated antenna exhibited omni-directional radiation as displayed in Fig. 9 (e).

![Figure 9. Fully printed antenna: (a) as-designed monopole antenna, (b) ground plane, (c) as-fabricated prototype, d) simulated vs measured reflection coefficient of the antenna, and e) simulated 3D radiation pattern.](image)

Conclusions

In summary, screen-printable AgNW inks were developed by tuning the AgNW loading, solvent compositions and polymer weight percentages. We showed that the resulting AgNW patterns were highly conductive as well as transparent. The high conductivity and transparency were achieved with a combination of washing cycles and photonic sintering. The washing cycle aided in enhancing the initial conductivity by removing the excess polymer from the printed films while maintaining their adhesive properties. The photonic sintering welded the NWs for increasing the number of conductive paths. Thus, the best tradeoff combinations of conductivity and transmittance, viz. ～5.48 × 10^8 S/m at 78 % and ～6.25 × 10^8 S/m at 83 %, were achieved. The printed patterns showed an excellent mechanical stability under 10,000 bending cycles. The printed films exhibited excellent electrical, optical and mechanical properties. In addition, an antenna was designed and fabricated, and the corresponding results were consistent with those obtained via simulations. The proposed transparent ink with excellent electrical and mechanical properties is promising for the realization of transparent and wireless passive electronic components.

Acknowledgements

The authors acknowledge the financial support provided by Ericsson with OSR #4606-EAB/DJN 2021-09-16. The authors acknowledge KAUST Core Labs facilities in the material characterizations. The authors also acknowledge Ms. Rebecca Esposito from Prof. Suzana Nunes’ lab in KAUST for her help in the viscosity measurements.

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