Contents lists available at ScienceDirect

Organic Electronics

journal homepage: www.elsevier.com/locate/orgel

Intense-pulsed-light irradiation of Ag nanowire-based transparent electrodes for use in flexible organic light emitting diodes

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ARTICLE INFO

Article history: Received 23 August 2014 Received in revised form 8 November 2014 Accepted 11 December 2014 Available online 22 December 2014

Keywords: Intense pulsed light Ag nanowire Organic light emitting diode Flexible electrode Transparent electrode

ABSTRACT

Silver nanowire (AgNW) based transparent electrodes are inherently coarse and therefore typically are only ever weakly bonded to a substrate. A remarkable improvement in the characteristics of a AgNW network film has, however, been achieved through a simple and short process of irradiating it with intense pulsed light (IPL). This not only avoids any severe deterioration in the optical characteristics of the AgNW film, but also significantly improves its electrical conductivity, adhesion to a polymeric substrate, and ability to endure bending stress. Most important of all, however, is the finding that the surface roughness of AgNW networks can also be improved by radiation. In a series of measurements made of organic light emitting diodes fabricated using these treated electrodes, it was revealed that the leakage current can be notably reduced by IPL treatment.

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1. Introduction

Silver nanowires (AgNWs) are considered one of the most promising candidates for replacing the indium tin oxide (ITO) that is conventionally used as a transparent electrode material; AgNWs providing both good optical and electrical performance, but with better stability when exposed to dynamic environments [1–5]. This is mainly supported by the anisotropy in their physical shape, with only a small fraction of percolated AgNW network being needed to form a highly conductive electrode. However, there are two major drawbacks that can arise from the

http://dx.doi.org/10.1016/j.orgel.2014.12.015

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direct coating of AgNWs onto a glass or polymer substrate: namely, a rough surface finish and poor substrate adhesion.

A rough AgNW network on a planar substrate is problematic in the sense that it can induce current leakage by providing a protrusion of AgNWs through the upper layers. Any such current leakage will limit the efficiency of the device, and may develop into a short circuit during its operational life time that can sometimes cause catastrophic device failure due to localized regions of high current density and the concomitant steep rise in temperature in these shorted regions [6]. The issue of poor adhesion to the substrate, on the other hand, makes it more difficult to fabricate fine patterns by photo-lithography and chemical etching. It should be noted here that transparent electrodes need to be very finely patterned in order to be of any use in high-resolution displays or narrow-bezel touch sensors. For example, today's touch panels have a line width and pitch





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smaller than 10 and 20 μ m, respectively, which means that printing and pattern transferring technologies cannot yet be employed industrially in their present form. Another disadvantage caused by the poor adhesion is the limited flexibility of the AgNW-network coating on the polymer.

In a bid to resolve these issues, there have been a number of different approaches reported. For instance, smooth transparent electrodes have been achieved by embedding AgNWs in the surface of a polymer matrix [7–11], wherein a liquid polymer is applied to a pre-coated AgNW network, then simply peeled off the supporting substrate once cured. Using this method, both solid and smooth conductive surfaces can be obtained by burying the AgNW film in the surface of the polymer matrix. However, owing to the fact that a large proportion of the stacked AgNWs remains buried beneath the surface of the polymer matrix, the surface coverage of the conductive paths is invariably limited and can result in the poor injection of carriers into the active layers. An alternative method is to make a conductive composite by mixing or stacking AgNW networks with other conductive materials such as graphene, carbon nanotubes or poly(3,4ethylenedioxythiophene) poly-(styrensulfonate) (PED-OT:PSS) [12–19]. It has been confirmed that this approach can produce a smoother surface; however, poor adhesion of the AgNWs to the substrate remains a major issue.

Attention has also been given to using photonic sintering as an effective means of enhancing the conductivity of metallic nanostructures without the need for thermal heating [20–22]. One such method, namely intense pulsed light (IPL), has been reported to be a powerful tool for enhancing adhesion by partially melting the regions of a polymer substrate that are immediately adjacent to AgNWs [21]. This revealed that only a very short irradiation of high-energy pulsed light is needed to induce the localized heating required to activate the interface between the metal and polymer. As a result, the polymer film absorbs practically none of the irradiated energy, and therefore is not severely damaged by this approach. Interestingly, these studies relied on using AgNWs that were only partially embedded into the surface of a polymer film; and thus, proper embedding of the AgNWs may allow the rough surface of the film to be sufficiently smoothed to allow its use as a transparent electrode in thin-film devices.

Building on this past work, IPL photonic sintering was employed in this study to produce smooth and solid AgNW networks on colorless-polyimide (cPI) substrates. For this, an AgNW film with an optimized density was fabricated by simple bar-coating as the bottom electrode of an organic light emitting diode (OLED), and was then irradiated with varying exposure times of 500–2000 µs. A yellow-emitting phosphorescent OLED was then fabricated on this AgNW electrode and a series of emission parameters, ranging from leakage current to the efficiency of emission, were measured in order to determine the effects of IPL irradiation.

2. Experimental procedure

2.1. Fabrication of the transparent electrode

The procedure used for the fabrication of the AgNWbased transparent electrode is schematically illustrated in

Fig. 1. In this, a glass substrate is first cleaned using detergent, de-ionized water, acetone, and isopropanol. A cPI varnish (Kolon, colorless-polyimide, Korea) is then spincoated onto the cleaned glass and subsequently cured at 200 °C for 1 h. The thickness of the film could be precisely controlled by varying the spinning velocity, and so a thickness of 20 µm was selected to provide a suitable balance of flexibility and good reliability. Next, the cPI-coated glass was placed on a Mayer rod apparatus and several drops (0.5 ml) of a nanowire solution (Nanopyxis Ltd., Korea) were deposited on it. Immediately afterwards, a Mayer rod #8 (R.D. Specialties, Inc., USA) was rolled over the drops to evenly spread the nanowire solution over the cPI surface. The resulting film was heated in an oven at 50 °C for 30 min to remove any remaining organic solvent from the coating layer.

A photonic sintering system (Polytec Ltd., Sinteron 2000, USA) was employed in the IPL irradiation (power input: 3 kV) of the samples, with the exposure time being varied from 500 to 2000 μ s. A PEDOT:PSS solution with a binding system (Dittotechnology Ltd., Korea) was then spin-coated at 1000 rpm onto the AgNW network to planarize its surface. After 30 min of curing in a convection oven at 120 °C, the PEDOT:PSS layer could be etched by an acidic solution (Chromium Etchant, Sigma–Aldrich). The resulting hybrid transparent electrode system consists of PEDOT:PSS and AgNWs, and as shown in Fig. 1, can be patterned simultaneously to its fabrication.

2.2. Evaluation of the transparent electrode

Scanning electron microscopy (SEM, JEOL Ltd., JSM6700F, Japan) was used to investigate the microstructure of the AgNW networks. Their optical transmission and haziness were also measured using a UV-visible spectrophotometer (Jasco, V-560, Japan), while the sheet resistance was measured by a non-contact measurement system (Napson Corporation, EC-80P, Japan). The surface morphology was measured by atomic force microscopy (AFM, Park Systems, XE-100TM, USA), and cross-sections of each sample were prepared by a focused ion beam (FIB, JIB-4601F, JEOL Ltd., Japan) system.

The mechanical reliability of the cPI film after being peeled from the glass supporter was evaluated using an adhesive tape (3 M, USA) test. This test was repeated 10 times for each sample to evaluate the adhesion of its AgNWs to the cPI film, and an automatic bend testing machine (Toyoseiki Ltd., MIT-DA, Japan) was used to measure the long-term reliability under repeated cycles of bending. This particular equipment caused the electrodes to alternately experience outward bending (tensile stress) and inward bending (compressive stress), ultimately resulting in cyclic fatigue failure. A bending radius of 0.5 mm was used to induce \sim 2% tensile and compressive strain. The electrodes were bent at a cycle rate of 1 Hz, and their resistance was measured during both the inward and outward bending cycles. More than 15 samples were fabricated and measured for various parameters, e.g., transmittance, haziness, sheet resistance and resistance with bending test.



Fig. 1. A schematic description of the fabrication process for AgNW/PEDOT:PSS-based electrode on cPI substrate.

2.3. Fabrication and measurement of OLEDs

The fabricated AgNW–PEDOT:PSS composite electrode was heated at 50 °C for 1 h in a vacuum oven to remove any residual water; after which, all of the organic and inorganic layers require to fabricate an OLED were deposited by thermal evaporation. The organic layers consisted of: (1) PNH-HT-1026 (P&H Tech Ltd., Korea) as a hole transporting layer (HTL), (2) PNH3008 as a bi-polar type singlet host and PNH3505 as a dopant to produce a yellow emitting layer (EML), (3) 2,2',2"-(1,3,5-benzenetriyl)-tris(L-phe-nyl-l-H-benzimidazole) (TPBi) as a hole blocking layer (HBL), (4) 4,7-diphenyl-1,10-phenanthroline (Bphen) as an electron transport layer (ETL). A final LiF/Al layer was then deposited to serve as the cathode.

The current–voltage (*I–V*) characteristics of the fabricated OLEDs were measured with an experimental set-up consisting of a Keithley 2400 source meter combined with a calibrated photodiode. All measurements and data acquisition were controlled by National Instrument's LabVIEW software. A spectroradiometer (Minolta, CS1000, Japan) was also employed to measure the electroluminescence spectrum of the $4 \times 6 \text{ mm}^2$ emitting area of the devices. For all test conditions, 10 samples were used to measure the device performances.

3. Results and discussion

We first investigated the effect of IPL exposure time on the characteristics of the AgNW-coated cPI film. For this, the power input was fixed at 3 kV, as no visible change in the optical or electrical parameters was found when testing was performed at lower inputs. Two different approaches were used: the first being to employ a single long-duration exposure, while the other was to use multiple, shorter exposures. Fig. 2 shows the effect of using a longer exposure (2000 µs) on the optical transmittance of the AgNW/cPI structure; revealing that although the change in transmittance is negligible, the haziness is increased by $\sim 0.4\%$ across a wide range of the visible wavelength. The inset in Fig. 2(a) also indicates that severe damage occurred at the edge of the film, which clearly means that a 2000 µs exposure is excessive for AgNW sintering. Note that the color of this damaged area changes from colorless to dark-brown, which is similar to the effects seen with high-energy irradiation of AgNW films reported in the literature [21]. Given that the aim here was to find the optimum sintering conditions for a smooth and solid AgNW/cPI film without causing any deterioration of its optical properties, the second approach was subsequently used (i.e., multiple instances of shorter singleexposure times, with a 10 s interval between each exposure).

Fig. 3(a) and (b) shows the effect of irradiation on the transmittance and haziness of the AgNW/cPI films. As mentioned, the use of multiple exposures prevents deterioration of the optical properties, which can be explained by the fact that the AgNWs have time to cool between each exposure. Interestingly, the effect of multiple exposures on bare cPI was quite different, with Fig. 3(d) showing a clear increase in its haziness by about 0.7% as the number of exposures is increased to eight. Given this, it is concluded that the effects of IPL irradiation differ depending on the materials that make up the film structure. This difference is believed to be mainly affected by two factors, i.e., high density of AgNWs covering the surface of cPI and higher thermal conductivity of AgNWs than polymer film (cPI). In case of AgNW coated cPI, the radiated energy is largely absorbed by AgNWs that are occupying about 10-20% of the cPI surface, and it converted to the heat energy for partial melting of the cPI beneath the individual AgNWs. The radiation to the rest of the surface area would affect the deterioration of the cPI, which is 10-20% smaller than the original film area, resulting in smaller damage of the film. Secondly, high thermal conductivity of Ag should



Fig. 2. Optical characteristics of the AgNW/cPI with and without IPL irradiation for 2000 µs: (a) transmittance spectrums, and (b) hazeness spectrums.



Fig. 3. Optical characteristics of the AgNW/cPI and bare cPI with and without multiple IPL irradiations for 500 µs: (a and b) spectrums for AgNW/cPI, and (c and d) spectrums for bare cPI.

have a role to dissipate the heat formed by the radiation, and therefore the raised temperature at the film surface could be cooled for a shorter time. Considering that the haziness of the polymeric film is affected by the formation of pores or other defects on its surface, the AgNWs coated on the cPI could make it delayed for deterioration of the film. When a bare cPl film is exposed to the radiation of IPL, the accumulation of thermal energy without fast dissipation can easily destroy it, even if the individual exposure times are short and separated by a period of cooling.

Fig. 4 shows the change in the sheet resistance of the films with an increasing number of 500 μ s exposures to

IPL irradiation; the decrease in sheet resistance observed in the case of a single 2000 µs also being shown for comparison. We see from this that the sheet resistance decreases with an increasing number of exposures, which can be mainly attributed to the plasmonic welding of the AgNWs and the removal of polyvinylpyrrolidone (PVP) from their surface. Many previous studies have reported that a thin layer of PVP remains on the surface of as-synthesized AgNWs, and that this affects the electrical conductivity of the AgNW network [1–5]. Thus, when an AgNW film is exposed to IPL irradiation, the AgNWs absorb much of the heat energy generated. This absorption of heat should also increase the temperature of any AgNWs nearby, which is the main force behind the elimination of PVP residue and the welding of AgNWs. Significantly, the results show that three short-term exposures of 500 µs each is more effective in reducing the sheet resistance than a single 2000 µs exposure. This confirms that multiple short-term exposures to radiation can be a more effective and safer way of fabricating highly transparent and conductive electrodes based on AgNW networks.

The tilt-view of AgNWs on cPI shown in Fig. 5 demonstrates that the as-deposited AgNWs have a porous structure, with visible gaps occurring between them and the cPI. Moreover, the edges of the wires are quite sharp, and there is no discernible evidence of the AgNWs being fused at their intersections. However, fusion of the cPI is observed when the number of exposures reaches four (as shown in Fig. 5(c)), and thus the gap between the wires and the substrate becomes filled with fused cPI. The gaps at the intersections of AgNWs are also no longer observed, which means that the surface of the wires is melted or diffused to form a solid network and explains the observed reduction in sheet resistance. It is interesting to note also that the surface of the cPI away from the AgNWs is still clear, and does not exhibit any obvious pores beneath it. After 8 exposures to IPL irradiation (Fig. 5(d)), the fusion of the cPI is intensified and results in a partial embedment of AgNWs into the cPI film. In this case, a large proportion of the wires is still exposed to air, and so provides sufficient contact with overlying materials such as PEDOT:PSS.



Fig. 4. Measured sheet resistance of AgNW/cPI films in accordance with the IPL radiation time and number.

Furthermore, as it is only the lower part of the wires near the interface with the cPI that is covered by the cPI, this can enhance the adhesion of the AgNWs to the substrate. In contrast, in the case of AgNWs transferred by coating a liquid polymer onto a pre-coated AgNW network, most of the nanowire bundles are buried beneath the surface of the cured polymer [9,10]. Consequently, only a small number of AgNWs can make contact with the upper material, resulting in a poor injection of carriers from them. According to a previous report, a high driving voltage and low current density are produced in a AgNW–polymer composite structure, which was related to a low surface coverage of AgNWs [10]. In Fig. 5(d) there is some evidence of pores being formed, but these are insufficient to affect the roughness of the electrode.

The surface roughness of the samples with and without IPL irradiation was measured by AFM, the results of which are shown in Fig. 6(a) and (c), respectively. From this, it is evident that with eight short IPL exposures of 500 µs, the difference in height from peak to valley (R_{pv}) and the root mean square (R_{RMS}) values decrease by about 30% and 50%, respectively. These figures indicate that the roughness of an AgNW film can only be improved by a suitable amount of photonic energy, which is supported by the partial fusion of the polymer substrate underneath the metallic wires and sintering of AgNWs. However, these roughness values are still too high to be suitable as a base for the fabrication of thin film devices, and so another planarization layer is clearly needed to further flatten the surface. For this purpose, we employed PEDOT:PSS: spin-coating it onto the AgNW networks as shown in Fig. 6(b) and (d). Although the same coating conditions were applied, a thinner and more planar layer was formed on the IPL-irradiated sample (Fig. 6(b)). By the AFM measurement for the overcoated PEDOT:PSS surfaces, it was observed that Rpv and R_{RMS} measured before IPL irradiation were 89 and 12 nm, while those for IPL-treated were 41 and 6 nm, respectively. This is attributed mainly to the more solid form of the AgNW networks on the cPI, as well as their reduced surface roughness.

The solidity of the AgNW networks on the cPI film was assessed by tape and bending endurance testing, as shown in Fig. 7. This revealed that the sheet resistance of the AgNW film without IPL radiation was increased to tentimes the value of its as-deposited state after just three repeats of testing, and yet did not vary at all up until the end of testing in the case of those samples exposed to IPL. The bending test results illustrated in Fig. 7(b) also show that the IPL-irradiated AgNW/cPI samples maintain their electrical performance after being subject to 10,000 bending cycles, whereas a catastrophic failure occurred after just 100 cycles in the samples without IPL exposure. Indeed, with the IPL-irradiated samples, the alternating inward and outward bending leaves the resistance virtually unchanged until at least 1000 cycles. Beyond this, the resistance does start to increase gradually and fluctuate at some points, but there is no steep increase in the resistance until 10,000 cycles. Furthermore, it was found that there is no evidence of cracking or plastic deformation in the cPI substrate. Consequently, it is believed that the



Fig. 5. Tilt-views taken by SEM for samples with increasing number of IPL exposure (500 µs): (a) a single exposure, (b) 2 times, (c) 4 times, and (d) 8 times.



Fig. 6. Top-view images taken by AFM and cross-sectional SEM images for samples with and without IPL exposure (500 us): (a and b) after 8 times of irradiation, (c and d) not-irradiated.



Fig. 7. Mechanical reliability of the samples with and without IPL exposure (8 times of 500 µs exposure): (a) measured sheet resistance with number of tape testing, and (b) measured resistance with bending cycles (bending radius: 500 µm).

increase in resistance is directly related to the weakening of AgNW contacts and the breaking of individual AgNWs.

To demonstrate the effects of IPL irradiation on light emission, yellow phosphorescent OLEDs with a structure of PEDOT:PSS/PNH2026/PNH3008:PNH3505/TPBi/LiF/Al were deposited on a AgNW/cPI substrate. With the exception of PEDOT:PSS, all materials required for the construction of this device were thermally evaporated consecutively. Fig. 8(a) and (b) shows the current density-bias voltage and luminance-bias voltage characteristics of the yellow-phosphorescent OLEDs based on AgNWs. The characteristics of these devices were measured to a voltage of 12.0 V, with a careful comparison of the two different cases (i.e. with and without IPL irradiation) presenting a couple of



Fig. 8. Measure parameters for fabricated OLEDs with and without IPL exposure (8 times of 500 µs exposure): (a) current density-bias voltage, (b) luminescence-bias voltage and (c) current efficiency-luminescence.

interesting points. Firstly, the current density-bias voltage curves show that the leakage current of the IPL-irradiated sample $(7 \,\mu\text{A/cm}^2)$ is lower than that of the non-irradiated one $(130 \text{ }\mu\text{A/cm}^2)$ by two orders of magnitude at a reverse bias voltage of 2.0 V. The higher leakage current of the non-IPL-irradiated samples is attributed to the rather coarse surface produced by their porous AgNW networks. In contrast, the improved surface smoothness created by partial embedment of the AgNWs within the cPI surface helps suppress their protrusion through the thin organic layer. Secondly, the current density above turn-on voltage is lower in those samples with IPL than without. It was noted that the contact area between AgNWs and PEDOT: PSS is reduced through partial embedment of the AgNWs after IPL treatment, thus implying that the surface coverage of the conduction paths is decreased slightly due to the fusion of the polymer. However, from a practical point of view, it could be said that the turn-on and injection properties are almost identical between the two cases. Moreover, the ${\sim}10\%$ decrease in current efficiency shown in Fig. 8(c) could also be regarded as being of little practical significance. Indeed, when taken in consideration with the excellent bending stability presented in Fig. 7(b), the results in Fig. 8 suggest that IPL irradiation can be used to make flexible OLEDs with improved stability under real-world driving conditions.

4. Conclusion

Through this study, it has been demonstrated that the mechanical and functional properties of AgNW networks can be significantly improved by using the radiation produced by a high-energy pulsed light. This addresses the current issue that although AgNWs have the potential to provide a highly conductive, transparent and flexible electrodes, their rough surfaces and poor adhesion has so far hindered their full-scale application in the fabrication of flexible OLEDs. This new approach allows for AgNWs with a significantly improved electrical conductivity, resistance to bending stress and adhesion to a polymeric substrate; and yet, does not severely impact on their optical characteristics.

Most important of all, however, is the fact that the surface roughness of AgNW networks on cPI can be reduced by this same treatment. This was demonstrated by the fact that a simple phosphorescent OLED based on a IPL-irradiated AgNW electrode has a greatly reduced leakage current (two orders of magnitude), with the loss of only 10% of its current efficiency. When this is combined with other improvements in various characteristics, it is apparent that IPL irradiation presents an effective solution for the fabrication of highly efficient and flexible OLEDs based on AgNWs.

Acknowledgements

This work was supported by the Technology Innovation Program (10042414, More than 60" transparent flexible display with UD resolution for IT fusion Infortainment System), as funded by the "Ministry of Trade, Industry & Energy (MI, Korea)".

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