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REVIEW

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Review on dry film photoresist-based patterning of Ag nanowire flexible electrodes for wearable electronics

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Abstract

Silver (Ag) nanowires have attracted significant attention as flexible electrodes for various wearable electronic devices owing to their excellent optical and electrical properties. Patterning is an important step in the fabrication of Ag nanowire electrodes of appropriate size and shape for electronic device applications. Among the various methods to pattern Ag nanowires, photolithography using a liquid photoresist is the most widely used. However, some factors have limited an extensive use of this method, such as the non-uniform thickness of liquid photoresist on large-scale coatings and the requirement of a post-annealing step that limits the application to few polymeric substrates. In turn, these factors affect the successful application of the roll-to-roll fabrication process. Dry film photoresist (DFR), a solid-state film-type photoresist, can enable a fast and simple patterning process at lower temperatures, as DFR-based patterning process only requires a simple lamination step to coat the photoresist on the substrates. The DFR process is performed at a temperature below 80 °C, which enables the application of most polymeric substrates. Furthermore, this process doesn't involve any additional post-annealing process, which makes it an appropriate technology for the roll-to-roll fabrication process. Owing to the advantages of DFR patterning, several recent studies have focused on this process for Ag nanowires patterning. This review provides an overview of successful examples of Ag nanowire patterning based on the use of DFR, along with the application of patterned Ag nanowires substrates as obtained by this method.

Keywords: Dry film photoresist, Patterning, Silver nanowire, Electrode, Flexible

Introduction

With the development of flexible/wearable devices, stable transparent electrode materials have attracted increasing attention (Hu et al., 2010; Kim et al., 2013b; Lee et al., 2016; Lee et al., 2014a; Yu et al., 2011). Currently, indium tin oxide (ITO) is the most widely used material for transparent electrodes owing to its high optical transmittance, conductivity, and excellent chemical stability. However, the mechanical brittleness of ITO-based electrodes has limited their application in wearable devices (Jiu et al., 2013; Lee et al., 2013; Lee et al., 2014b). Therefore, research has been actively conducted to investigate the application of graphene, carbon nanotube (CNT), metal mesh, and metal nanowires

as flexible transparent electrodes (Hu et al., 2011; Rowell et al., 2006; Wang et al., 2008; Zhang et al., 2006). Among them, metal nanowires have attracted particular attention as next-generation transparent electrode materials owing to their high visible light transmittance and conductivity, which are comparable to those of ITO. Particularly, Ag nanowires have emerged as the most promising candidate for flexible transparent electrodes owing to their scalable synthesis by a solution-based polyol process and their ability to coat large-scale substrates through a roll-to-roll technology (Hwang et al., 2014; Kim et al., 2013a; Lee et al., 2008). However, in order to obtain effective Ag nanowire electrodes for integration in flexible devices, it is essential to address main aspects, such as the chemical and mechanical reliability of Ag nanowires, and the development of an appropriate patterning technology.

Photolithography using a liquid photoresist (PR) is the most commonly used patterning technology for Ag nanowires (Kim et al., 2018). During this process, Ag nanowires are coated with PR via spin coating, followed by thermal baking at ~ 100 °C. Subsequently, the PR is selectively hardened or softened via exposure to ultraviolet (UV) light using a photo-mask, after which it is retained on the substrate or removed by placing it in a developing solvent. Thereafter, the PR is subjected to an etching step, during which the Ag nanowires under the PR are protected from the etchant, while the uncovered Ag nanowires are exposed to the etchant and therefore removed. Lastly, the remaining PR on the Ag nanowires is removed using a stripping solution, leaving only the patterned Ag nanowires on the substrate. As previously mentioned, the photolithography process using photosensitive liquid PR has been widely applied and related processing conditions and patterning tools have been well defined, even in industries. Nowadays, liquid PR-based patterning can be applied in industries without additional efforts to optimize the process and without any additional cost for new equipment. However, owing to the requirement of a heat treatment (~ 100 °C) step to harden the PR, the selection of an appropriate polymer substrate has limited the effective application of the liquid PR-based patterning process so far (Shaw et al., 1997). In addition, the thermal baking process and the use of only polymeric substrates affect the compatibility of the liquid-based PR with the roll-to-roll fabrication process (An et al., 2017). Laser patterning is an alternative method for patterning Ag nanowires in industries (Oh et al., 2016). Compared to photolithography, which involves various steps to achieve the formation of a pattern, laser etching can achieve the patterning of Ag nanowires in a single process, and can form very fine patterns. However, the slow patterning speed of lasers increases the processing time and the overall cost. Moreover, the laser can potentially damage the substrate owing to the high energies employed. All the above limitations have hindered the application of laser patterning of Ag nanowires, particularly aimed at an industrial production.

To overcome the limitations of existing patterning techniques, various researchers have developed new patterning methods for Ag nanowire electrodes (Kim et al., 2015; Liu et al., 2016; Song et al., 2015, 2016). For example, Song et al. reported the use of intense pulsed light (IPL) for patterning metal nanowires (Song et al., 2016). This is achieved by selectively irradiating Ag nanowires using IPL by ultra-high energy to enable the adherence of the Ag nanowires to the polymer substrate in the IPL-treated area (Fig. 1). The non-treated Ag nanowires exhibit a weak adhesion to the substrate and can

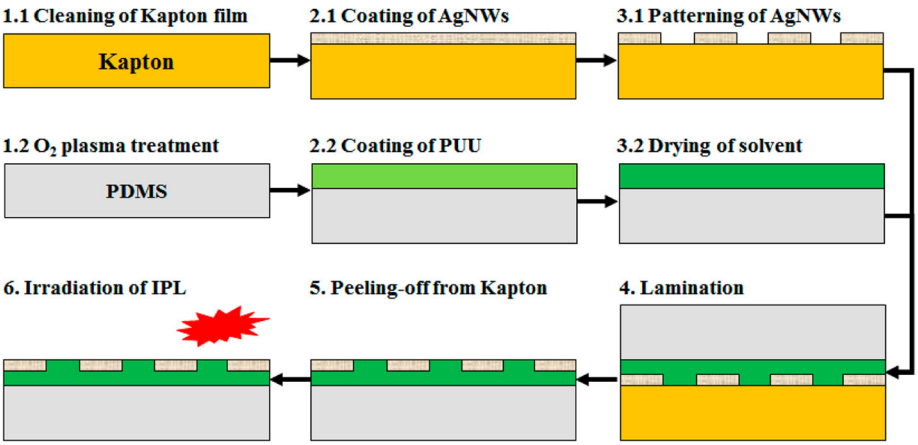

Fig. 1 Scheme of the fabrication procedure of patterned Ag nanowire electrodes based on IPL. Reproduced with permission from Song et al., (2016). Reprinted with permission from Song et al., (2016). Copyright 2015 American Chemical Society

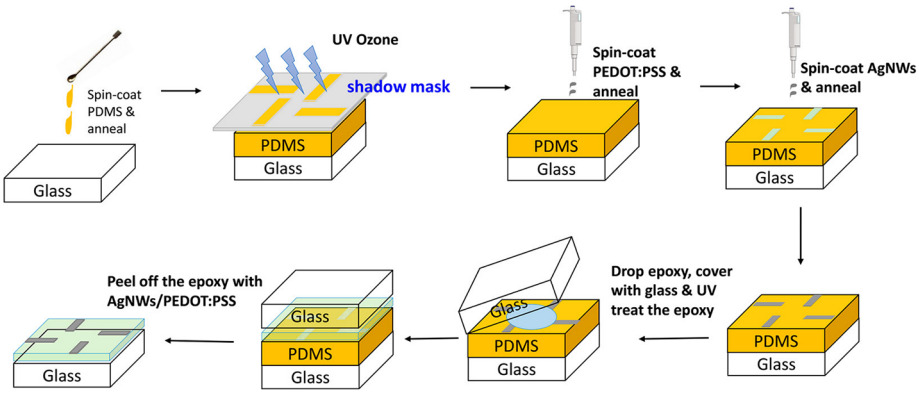

Fig. 2 Scheme of the fabrication procedure of patterned Ag nanowire electrodes based on UV-ozone treatment. Reproduced with permission from ref. (Liu et al., 2016). Reprinted with permission from ref. (Liu et al., 2016). Copyright 2016 American Chemical Society

be easily removed by mechanical wiping or ultrasonic cleaning, thus enabling the formation of patterned Ag nanowires on the IPL-treated area. Particularly, the IPL-based patterning technology has attracted attention owing to its easiness and fast patterning time. However, the potential risk of photothermal damage of the substrate has limited the further application of this method. On the other hand, rough removal steps, such as mechanical wiping or ultrasonic cleaning processes, limit the formation of uniform fine patterns over a large area. Liu et al. formed Ag nanowire patterns using a selective UV-ozone treatment method (Liu et al., 2016) by selectively conferring a hydrophilic nature to the hydrophobic surface of the substrate, after which Ag nanowires were selectively deposited to form patterns (Fig. 2). Although this process is simple and does not require a high-energy device that can damage the substrate, the resolution of the patterning was poorer than those of the previously reported patterning technologies (Kim et al., 2018; Li et al., 2018; Song et al., 2020). Considering that industries are mostly equipped with photolithography systems, the requirement of the installment of new equipment and the

significant efforts required to stabilize new processes significantly increase the production cost of newly developed patterning technologies. Therefore, to develop an effective patterning technology for Ag nanowire electrodes, the technology should be capable of utilizing existing photolithography facilities.

To solve the limitations of previously reported techniques, a patterning technique utilizing a dry film photoresist (DFR) has been reported (An et al., 2017; Kim et al., 2017b). DFR is a solid-state film-type photosensitive resin that can be coated through a simple lamination process. In addition, DFR-based process does not require any thermal baking process as instead required for liquid PR. Thus, it enables the use of various polymeric substrates compared to the liquid PR-based process. In addition, thanks to its solid state, it is possible to impart DFR with a uniform thickness on large-scale substrates, and to deposit it on peculiar substrates, such as holey polymeric films or cylindrical substrates. Particularly, existing photolithography facilities apart from a simple laminator can be employed to perform a DFR-based patterning process, which thus can be directly adapted to the industrial production lines. Because of these advantages, several studies have dealt with the use of DFR for Ag nanowire pattering [ref]. In this review, we summarize the patterning methods of Ag nanowire transparent electrodes based on DFR and discuss the applications of the as-obtained electrodes.

Patterning method of Ag nanowire electrodes using DFR

Figure 3 shows a schematic illustration of the patterning of Ag nanowires using DFR (An et al., 2017; Kim et al., 2017b). First, a DFR film is coated on a substrate coated with Ag nanowires using a simple lamination method at approximately 80 °C, in such a way that the thermal damage to the Ag nanowires during the process is regarded as negligible. Next, the DFR-coated Ag nanowire electrode is selectively exposed to UV light through a photomask for patterning, during which the UV-irradiated area is cured and remains

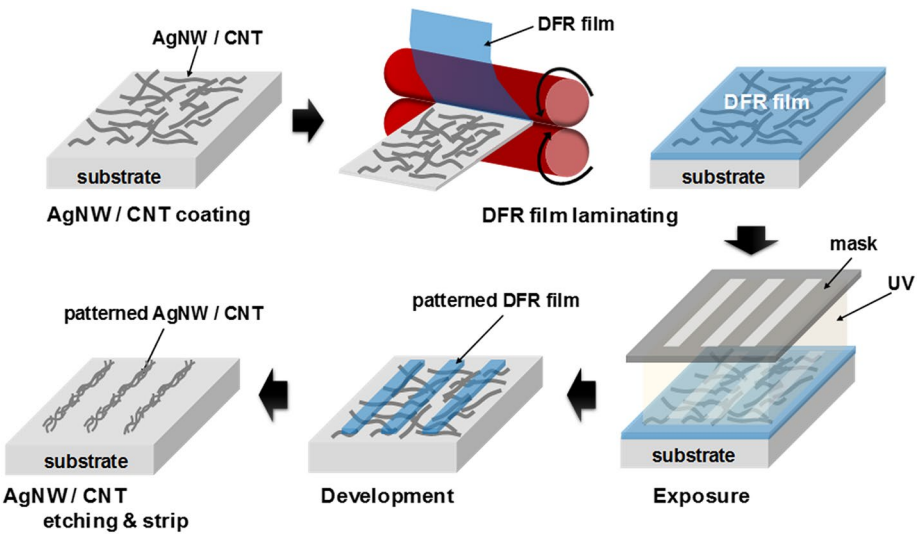

Fig. 3 Scheme of the patterning process using a DFR. Reproduced with permission from ref. (An et al., 2017). Copyright (2017), with permission from Royal Society of Chemistry

undissolved in the subsequent development process. For the development process after the UV treatment, the sample is immersed in a basic sodium carbonate (Na_2CO_3) aqueous solution, during which the uncured DFR is reacted with acrylic acid to solubilize the polymer that dissolves into the developer during the following developing process. The developing solvent can be applied using an air spray. The chemical reaction that occurs during the developing process can be represented as follows:

$$Na_2CO_3 \leftrightarrow Na^+ + NaCO_3$$

$$NaCO_3^- + H_2O \leftrightarrow NaHCO_3 + OH^-$$

$$DFR{-}COOH + Na^+ + OH^- \rightarrow DFR{-}COONa + H_2O$$

Next, the Ag nanowires that are exposed after the developing process are etched using a silver etchant. Subsequently, the etched Ag nanowires are immersed in an aqueous sodium hydroxide (NaOH) solution with a stronger alkalinity than Na_2CO_3 to strip the hardened DFR that is retained on the patterned Ag nanowires on the substrate. As the developing and stripping process are performed in alkaline aqueous solutions, the DFR method is considered to be less harmful compared to the liquid PR process that uses organic solvents.

Figure 4 shows a scanning electron microscopy (SEM) image of Ag nanowires and carbon nanotube (CNT) electrodes patterned using DFR-based method. In the case of CNTs, the patterns are formed using a plasma etching process owing to the difficulties in achieving CNT patterning by using a solution-based etching. Typically, down to 30 μm-wide patterns can be formed in both the Ag nanowires and CNTs over large-area substrates of 10 cm or above. Furthermore, a recent study confirmed the possibility of forming 10 μm-wide patterns (Fig. 4c internal picture), although this can only be achieved on a substrate with a size of less than 1 cm owing to the weak adhesion between

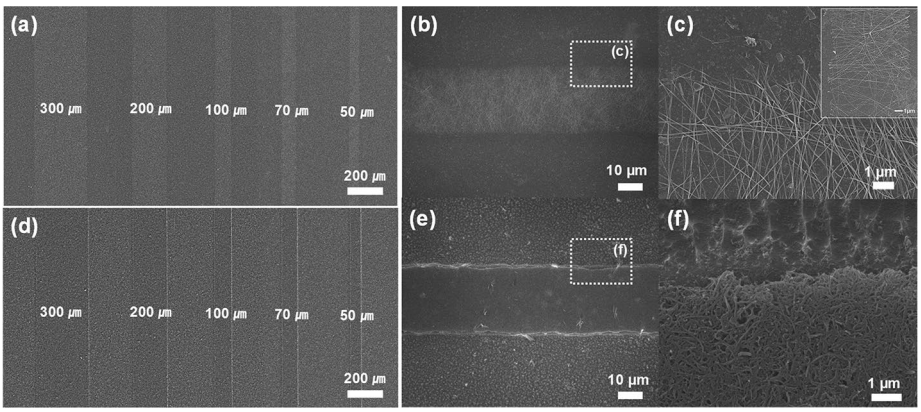

Fig. 4 Scanning electron microscopy (SEM) images of **a**–**c** patterned Ag nanowire networks and **d**–**f** CNT networks with different widths. **b** Patterned Ag nanowire networks and **e** CNT networks with 30-μm width. **c**, **f** High-magnification SEM images of the areas enclosed by the white squares in **b** and **e**, respectively. Reproduced with permission from An et al., (2017). Copyright (2017), with permission from Royal Society of Chemistry

Ag nanowires and substrate, which results in the removal of Ag nanowires during the stripping process of DFR. Anyway, 30 μm appears as an acceptable pattern accuracy for electrodes to be applied in sensors, such as touch panels, while applications requiring a smaller patterning are limited to small devices such as thin-film transistor electrodes, where, however, the 1 cm substrate size is sufficient.

Figure 5 shows the line resistance and the sheet resistance values of the transparent electrode as a function of the pattern size. As shown in the image, line resistance values increase as the Ag nanowire pattern size decreases, and there is a linear relationship between pattern size and line resistance value on the log scale, in accordance with the well-known percolation theory. In addition, the sheet resistance value calculated using the resistance value per unit area exhibited a similar value regardless of the pattern size, which is also consistent with the percolation theory. This demonstrates that the DFR-based patterning process induces minimal damage to the Ag nanowires, enabling pattern formation without significant deterioration in the electrical properties of the electrode.

It is worth noting that in the DFR-based patterning (Fig. 6), coating can be performed without subsequent heat treatment and using a simple lamination process, in turn resulting compatible with the roll-to-roll process.

Furthermore, a previous study confirmed that patterning can be performed without using an etchant through a lift-off method (Kim et al., 2017b) as shown in Fig. 7. In this case, compared to the afore-mentioned DFR process, the DFR on the Ag nanowires to be removed are not cured during UV exposure, whereas that on the Ag nanowires not to be removed are cured, which results in a strong adhesion to DFR of the latter. Subsequently, the cured DFR is removed using a stripping solution, along with the adhered Ag nanowires, finally obtaining down to 50 μm-wide Ag nanowire patterns (Fig. 8). A further decrease in pattern size results in an unavoidable hardening of the part that should not be cured, which affects the formation of a clean pattern. Owing to the pattern size limitations, this method can only be applied in specific applications requiring relatively large pattern size, such as touch panels or flexible circuit boards. Figure 9 shows a flexible organic light emitting diode (OLED) successfully

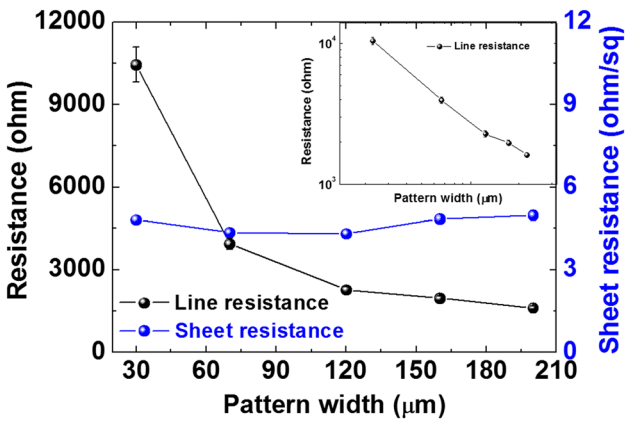

Fig. 5 Change in resistance and calculated sheet resistance as a function of pattern widths. The inset shows the logarithmically rescaled results of the line resistance. Reproduced with permission from An et al., (2017). Copyright (2017), with permission from Royal Society of Chemistry

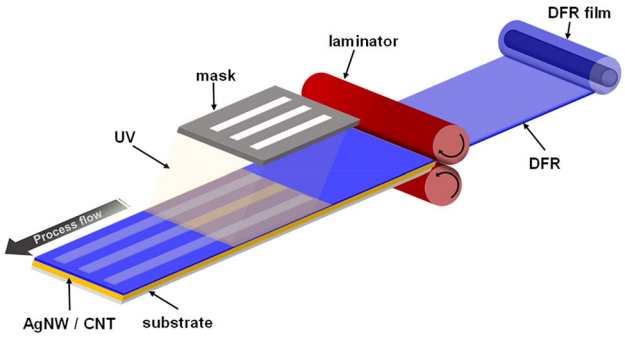

Fig. 6 Conceptual illustration of the roll-to-roll manufacturing system combined with the laminator for DFR-based patterning. Reproduced with permission from An et al., (2017). Copyright (2017), with permission from Royal Society of Chemistry

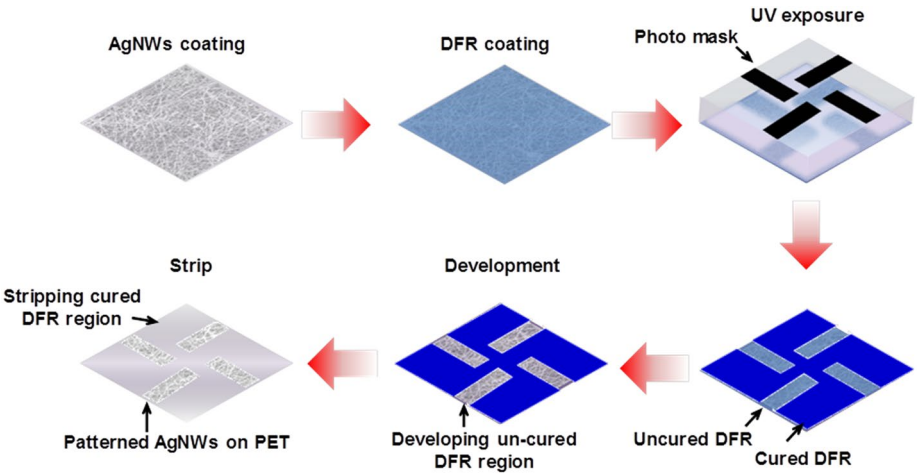

Fig. 7 Scheme of the etchant-free patterning and embedding of Ag nanowires according to a lift-off based DFR process. Reproduced from Kim et al., (2017b). Copyright (2017), with permission from Elsevier

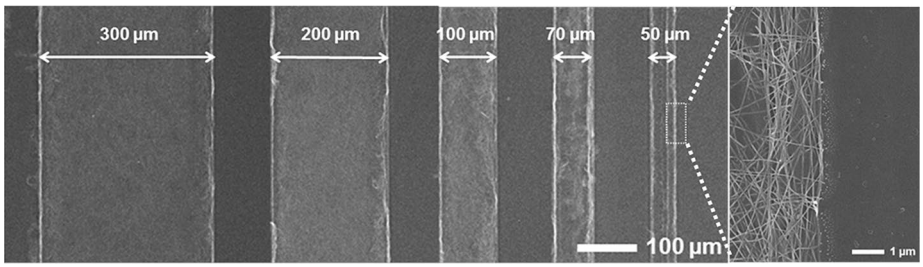

Fig. 8 SEM image of the Ag nanowire electrodes with different pattern widths, and the enlarged image of the white dot square. Reproduced from Kim et al., (2017b). Copyright (2017), with permission from Elsevier

fabricated using Ag nanowire electrodes patterned with the lift-off-based DFR process. The light emitting performance of the OLED fabricated using the patterned Ag

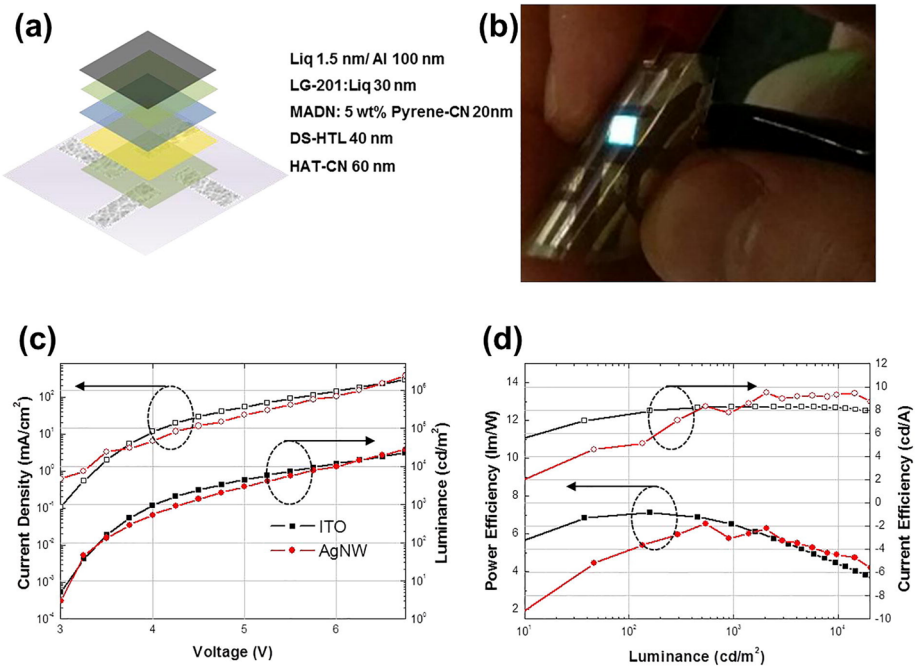

Fig. 9 Ag nanowires-based OLED. **a** Schematic structure of the OLED. **b** Operation of the OLED at 5 V. **c** Current density and luminance, and **d** power efficiency and current efficiency of the Ag nanowires-based OLED as compared with an indium tin oxide (ITO)-based OLED. Reproduced from Kim et al., (2017b). Copyright (2017), with permission from Elsevier

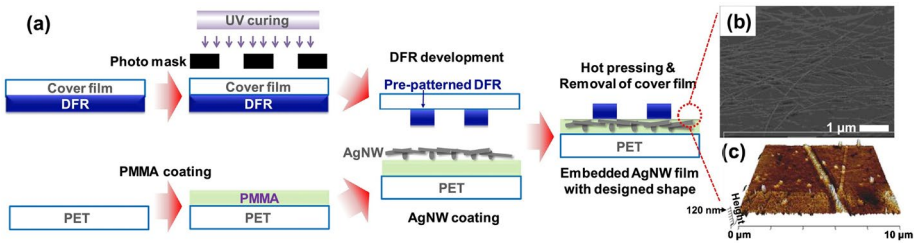

Fig. 10 **a** Schematic illustrations of the fabrication steps of patterned and embedded Ag nanowire electrodes with a DFR insulating layer. **b** 35°-tilted SEM image and **c** atomic force microscopy (AFM) image of PMMA-embedded Ag nanowires. Reproduced from Kim & Hwang, (2018). Copyright (2018), with permission from Elsevier

nanowires was comparable to those fabricated using ITO electrodes, confirming the feasibility of the lift-off-based DFR process for Ag nanowire patterning.

The patterned DFR can also be used as the insulating layer of Ag nanowire electrodes for flexible shaped OLEDs applications (Kim & Hwang, 2018). Figure 10 shows the fabrication process of patterned electrodes using DFR. First, the DFR is selectively cured using a photo mask with desired shape. The optical and SEM images of patterned DFR are shown in Fig. 11. Next, the selectively-cured DFR is hot pressed onto the Ag nanowire electrodes to form vertically-insulated Ag nanowire electrodes with desired pattern shapes. Lastly, the flexible OLED can be obtained by depositing an organic light emission layer on the DFR-shaped Ag nanowire electrodes (Fig. 12a and

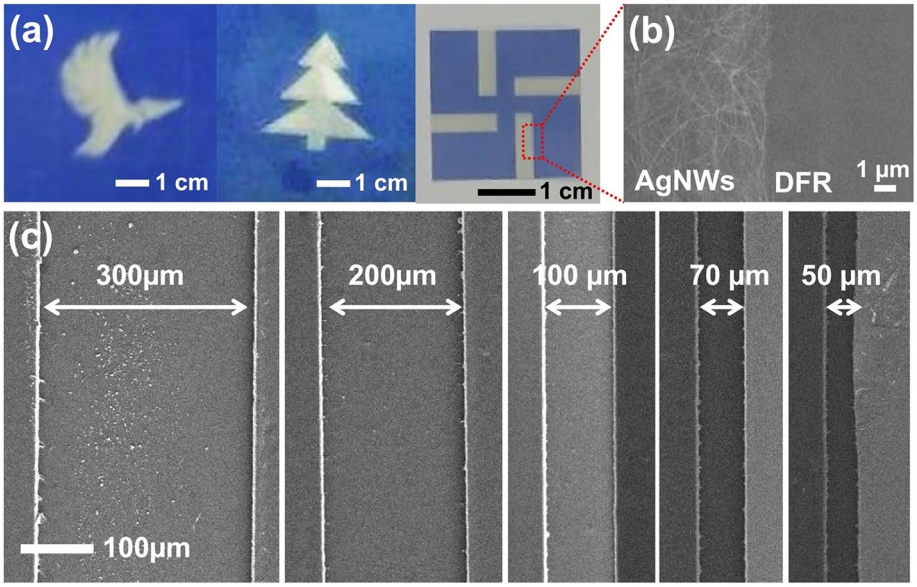

Fig. 11 **a** Photographs of pre-patterned DFR with various shapes attached to the AgNW/PMMA/PET electrodes. **b** SEM images of the edge between Ag nanowires and DFR, and **c** pre-patterned DFR with different line widths. Reproduced from Kim & Hwang, 2018). Copyright (2018), with permission from Elsevier

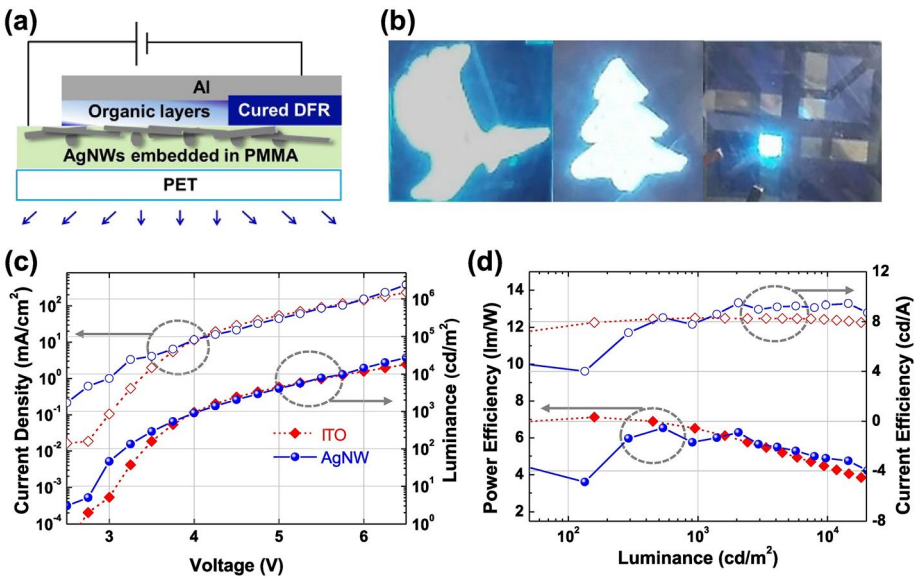

Fig. 12 **a** Schematic structure of a DFR insulator-based OLED. **b** Photographs of OLED fabricated using Ag nanowire/DFR electrodes with different shapes. Electroluminescence performances of Ag nanowire/DFR-based and ITO-based OLED: **c** current density and luminance, and **d** power efficiency and current efficiency. Reproduced from Ref. Kim & Hwang, (2018). Copyright (2018), with permission from Elsevier

b). The fabricated OLED exhibits high current densities and power efficiency values, which are comparable to those with ITO electrodes (Fig. 12c and d).

With an increase in the interest in eco-friendly products, paper-based electronics have attracted increasing attention. DFR can be used as a medium for transferring Ag nanowires to paper-based electronics (Kim et al., 2017a) as displayed in Fig. 13. First,

Ag nanowires are coated on a no primer-treated polyethylene terephthalate (NP-PET), which is then laminated with DFR. The Ag nanowires are embedded in the DFR during the lamination process. Next, the DFR-coated Ag nanowires on NP-PET are placed on a toner printed paper, which is hot pressed under 30 MPa at 90 °C, resulting in the separation of DFR-coated Ag nanowires from NP-PET and their transfer the paper. The feasibility of the transfer process is a consequence of the weaker adhesion of Ag nanowires to NP-PET as compared to paper. The DFR-transferred Ag nanowires exhibit good mechanical stability during 1000 bending cycles at a bending radius of 5 mm (Fig. 14a and b). In addition, a paper-based heater fabricated by the above method exhibits a homogenous heating ability, as shown in Fig. 14c, d.

Conclusions

In this review, the patterning method of Ag nanowire flexible electrodes using DFR is discussed together with its optimization and exploitation within different applications. Although many efforts have been devoted to ensure a commercialization of Ag nanowire electrodes, the development of an affordable patterning technology of Ag nanowires still represents an important challenge. Although standard photolithography processes or laser-patterning technologies can be used for patterning, it remains critical to develop an alternative technique showing compatibility with the roll-to-roll production process over large areas and aimed to further extend the application of Ag nanowires. Currently, the DFR-based patterning process exhibits various advantages owing to its convenience, rapidity, and simple deposition using lamination without need of a subsequent heat treatment. The compatibility of DFR with the roll-to-roll production process represents another advantage in the production of Ag nanowire electrodes. With these premises, the application of DFR-based patterning is expected to gather increasing interest in the immediate future, which can accelerate the commercialization of Ag nanowire flexible electrodes.

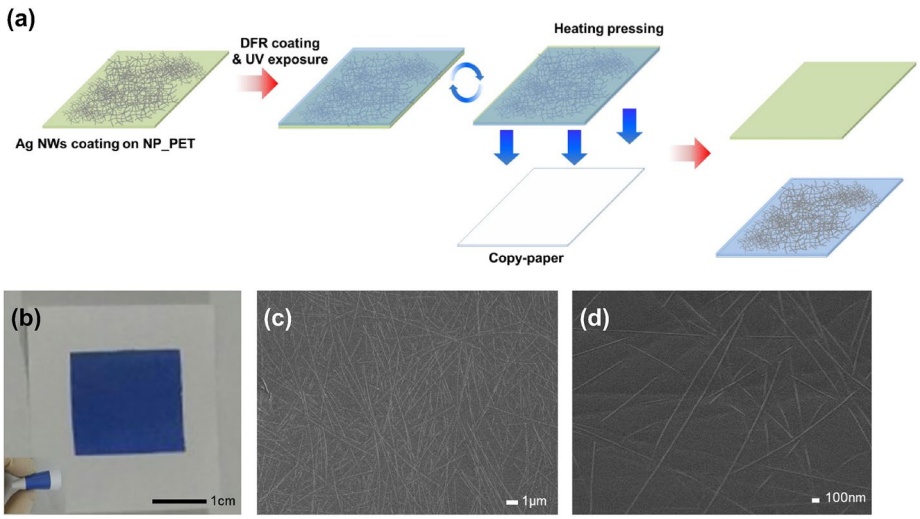

Fig. 13 **a** Scheme of the fabrication process of Ag nanowire/DFR/paper electrode. **b** Image of the Ag nanowires/DFR/paper electrode. The inset of **b** shows a bent Ag nanowire/DFR/paper electrode. **c** Plan-view and **d** 30°-tilted SEM images of DFR-embedded Ag nanowires. Reproduced from Kim et al. (2017a). Copyright (2017), with permission from Elsevier

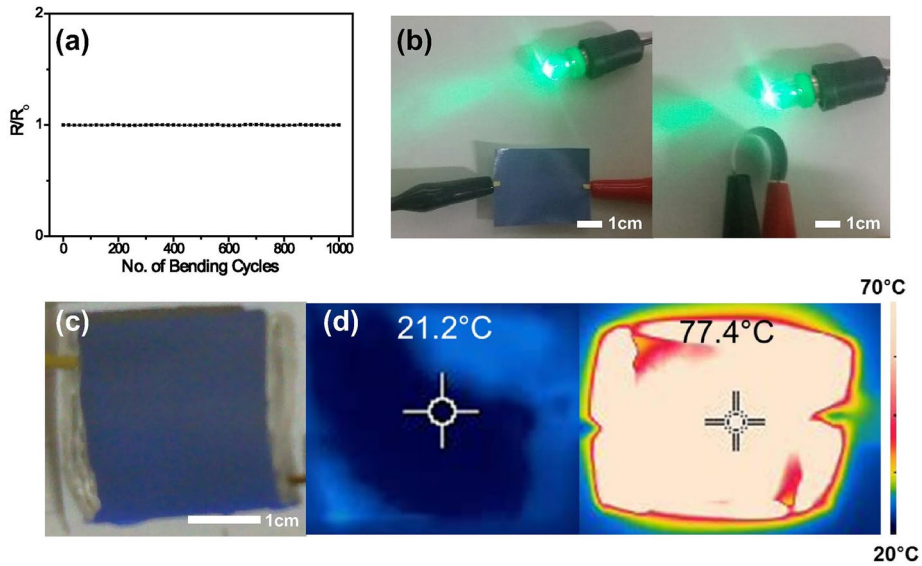

Fig. 14 **a** Change in resistance of Ag nanowire/DFR/paper electrode as a function of the number of bending cycles. **b** Images of the operation of a LED connected to an Ag nanowire/DFR/paper electrode before and after bending. **c** Image of the Ag nanowire/DFR/paper electrode connected to a power supply, and **d** IR images of **c** before and after imposing a voltage of 3.0 V. Reproduced from Kim et al. (2017a). Copyright (2017), with permission from Elsevier

Acknowledgements
Not applicable.
IRB permission is not applicable because there were no human or animal related experiments in this study.

Author contributions
BH and PM drafted manuscript. Both authors read and approved the final manuscript.

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Funding
This research was supported by the National Research Foundation (NRF) of Korea, which is funded by the Ministry of Science, Information and Communications Technology (Grant Nos. NRF-2019K1A3A1A25000230 and 2021R1F1A1054886).

Availability of data and materials
The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests
The authors declare that they have no competing interests.

Received: 3 March 2022 Accepted: 6 May 2022
Published online: 05 September 2022

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