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Flexible Transparent Electrode Materials and Their Application Progress in OLEDs

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Abstract. Organic light-emitting diodes (OLEDs) are an essential aspect of the development of unconventional devices. A flexible transparent electrode (FTE) is an essential component for OLEDs. However, the development of FTE is not always successful. Previously, scientists successfully developed the original indium tin oxide (ITO)-based electrodes; these electrodes displayed excellent properties. However, traditional ITO electrodes still have drawbacks such as brittleness and high cost. To resolve the previous problem, new strategies have been proposed to replace ITO-based electrodes in FTE. Emerging strategies focus on organic conductive polymers and metallic materials. In this review, the first strategy will mainly focus on development related to PEDOT: PSS; the basic properties of this polymer will be discussed, and some essential related techniques will be discussed. On the other hand, in this review, the discussion of metallic material-based FTE will focus on several aspects, including nanowires, ultra-thin metal membranes, and metal networks. After discussing them separately, the comparison between the two strategies will be made. As a result of the review, future research should not focus on using one material to substitute the ITO-based electrodes, and appropriate heterostructures should be designed to achieve decoupled control of conductivity and mechanical properties. This review also concluded the limitations of current research on FTEs and provides a reference for the development of more cost-efficient, better-performing, and more stable FTEs that can be used for unconventional display devices.

Keywords: Flexible transparent electrodes, wearable devices, organic light-emitting diodes, unconventional displays

1. Introduction

The fast advancement of flexible electronic devices, wearable electronics, and unconventional displays has imposed increasingly stringent demands on the performance of organic light-emitting diodes (OLEDs). The performance of conventional OLEDs tends to be restricted by rigid glass-based structures, and to successfully develop those unconventional forms of devices, each functional layer should withstand repeated folding and stretching without breakage, and their electronic properties should not deteriorate after experiencing deformations. To address those issues, transparent and flexible electrodes have been proposed with some new approaches, but they also become the core bottleneck for those problems, because new electrodes should satisfy three essential

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and seemingly contradictory standards: low sheet resistance, high optical transmittance, and excellent mechanical flexibility under cyclic strain.

On the other hand, the mainstream solution for OLEDs also has limitations. Previously, indium tin oxide (ITO) electrodes have served as the gold standard for the manufacturing of transparent electrodes because of their mature fabrication process and balanced optoelectrical properties. However, ITO electrodes are not suitable for implementation scenarios such as transparent flexible electrodes. Firstly, because ITO belongs to ceramic materials, its microscopic structure is hard and brittle, leading to microcracks upon elongation and folding. Consequently, the resistance of the electronic component will increase after just folding a hundred times. Furthermore, due to the scarcity of indium, the cost to manufacture an ITO electrode has risen. This is combined with the high-vacuum and high-temperature deposition process required for the fabrication of ITO electrodes. Those defects are incompatible with the goal of low cost and roll-to-roll continuous manufacturing. Those defects forced researchers to find a substitute for ITO electrodes [1].

Currently, the manufacturing of electrodes without ITO is based on two material branches; these two branches have intrinsic differences. The first approach is the conductive compound, representative of PEDOT: PSS; this material can be directly manufactured through aqueous solution, and this type of material has excellent flexibility. Those properties made this material an ideal candidate for the manufacturing of flexible electronic components using a purely solution-based process. The other type of material is metal-based electrodes, which include metallic nanowires, metallic networks, and ultra-thin metallic membranes. Owing to the high conductivity of metals, as the thickness of the membrane is reduced to the nanometer level, the membrane still has low sheet resistance. Now, both approaches have achieved some breakthroughs, but there are still some defects related to those approaches. PEDOT: PSS can easily absorb moisture from surrounding and it is acidic. In comparison, metallic nanowires have disadvantages such as high junction resistance and high surface roughness. Those disadvantages really became obstacles for the implementation of those approaches.

Although abundant articles exist on these two types of materials, few reviews have systematically addressed the integration of OLED components and summarized methods to improve the properties of compound and metallic nanowires. In the article, a review of current strategies that are widely considered, which include metallic and nonmetallic strategies, will be provided. For the nonmetallic strategy, which is widely regarded as a representative ITO substitute, the advantages and disadvantages of this polymer will be systematically reviewed, along with optimization strategies, fabrication techniques, and implementation examples. Similarly, the review of metallic strategy, include advantages and limitations, and implementation will be provided. In the end, the prospect of flexible transparent electrode (FTE) in the OLED field will be provided to find better strategies for developing better FTE and more opportunities for implementation.

2. Conductive compound and FTE

2.1. PEDOT: PSS: structural characteristics and intrinsic limitations

2.1.1. The structure of PEDOT: PSS

PEDOT: PSS is a composite organic structure that is used for a transparent flexible electrode. PEDOT means poly(3,4-ethylenedioxythiophene), and this is a type of conductive polymer that can be used in the OLED. In the implementation, PEDOT will not be used individually; it will be used at the same time as its counterion, such as PSS. The EDOT, which is the basic unit of PEDOT, is

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composed of a central five-membered thiophene ring fused with two six-membered dioxane rings. The geometric structure of the PEDOT molecule can be seen in Figure 1 below, and Figure 2 shows the molecular structure of PSS. This geometric structure increases molecular stability; moreover, the oxygen atoms increase electron density, which confers special conductivity to the molecule. In comparison, the PSS molecule has a much simpler structure with a backbone composed of styrenesulfonate, and on this backbone, some functional groups such as sulfate will be attached to this linear backbone. This PSS molecule served as a counterion of PEDOT and formed an essential conductive organic compound because of the different charges of PEDOT and PSS.

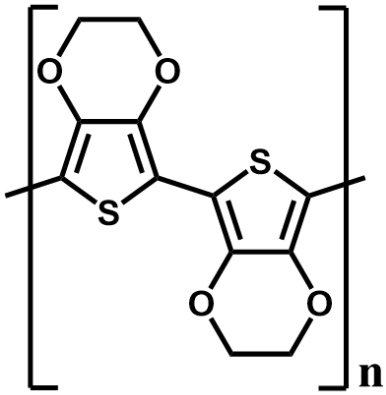

Figure 1. The molecular structure of PEDOT

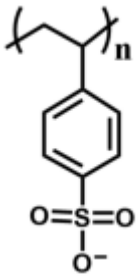

Figure 2. The molecular structure of PSS

2.1.2. Limitation of PEDOT: PSS

Even though this conjugated polymer has excellent properties, it still has some limitations. Its inhomogeneous electrical and morphological properties lead to poor long-term stability and shorten its shelf life [2]. Because this molecule has a rigid physical structure, it has low intrinsic stretchability. As this polymer is rigid, it can only be elongated by 2% to 5% when stretched, which means the polymer structure will easily break if force is applied [3]. This disadvantage limited the implementation of PEDOT: PSS in the field of flexible and wearable electronics such as flexible transparent electrodes. In addition, due to the presence of a sulfonic acid group in PSS, the product of dispersion of PSS is highly acidic. It creates a pH of around 1 [4]. After this highly acidic polymer is applied, the surrounding conductive oxides will be corroded, and it will also threaten other surrounding materials; as a result, the overall lifetime will be reduced. Another defect related to the performance of the material is its hydrophilicity. Because the PSS component is highly hydrophilic, it gradually absorbs moisture from surrounding environments. As a result, the absorbed water molecules will modify the initial molecular matrix of the molecule, and the conductive

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PEDOT chain will gradually become isolated from PSS components. This process is irreversible degradation for the material and seriously influences the conductivity of the material. Furthermore, due to its molecular structure, the intrinsic conductivity of undoped PEDOTLPSS remains relatively low, which is another disadvantage. One last limitation is the degradation of the molecule at high temperature (approximately 250 degrees Celsius). This issue is related to PSS, as it will chemically decompose at high temperature and the original performance of the material destroyed.

2.2. Strategy to improve PEDOT: PSS

To really deal with so many issues related to PEDOT: PSS and develop this advanced polymer, some strategies have been developed.

Firstly, one essential aspect is optimizing the conductivity of the polymer. Because the PSS structure surrounding the PEDOT core is insulating, the original PEDOT: PSS had low conductivity. The first strategy is adding polar solvent, such as Dimethyl Sulfoxide (DMSO), *N*, N-dimethylformamide (DMF), tetrahydrofuran (THF), or ethylene glycol (EG), to the PEDOT: PSS solution. Since these polar organic solvents have high boiling points, when applied as a post-deposition treatment, they can reduce the electrostatic attraction between positively charged PEDOT and negatively charged PSS. Consequently, the insulating PSS domains have a smaller impact on the conductivity of the polymer [5]. And then there is one subsequent effect: excess PSS will be removed after the solvent is applied. At the same time, conductive PEDOT can penetrate the PSS-rich regions, facilitating more efficient coupling of the PEDOT-rich domain and thereby enhancing the overall conductivity. At the same time, the number of π–π stacking interactions is also enhanced, and those interactions hold crystals closer and make the polymer more conductive.

Secondly, even though the conductivity of PEDOT: PSS can be optimized through the process of modifying intrinsic properties, there are still limitations. Because of the intrinsic rigidity of the conjugated pair of the polymer backbone, the rate of elongation is limited to around 2% to 5%, which limits its compatibility with the implementation of flexible electronics. Currently, one mainstream strategy is mixing an aqueous mixture of PEDOT: PSS with highly extensible insulative polymers to form a phase-separated or interpenetrating network. The materials that can be used for this optimization include waterborne polyurethane (WPU), polyethylene glycol (PEG), and polyvinyl alcohol (PVA). Nonionic WPU is very compatible with the PEDOT: PSS molecule, and it will not change its original property. Then, WPU will experience hydrogen bonding and coulombic attraction with PEDOT: PSS, and because this elastomer has advantages such as high deformability and can be combined with PEDOT: PSS through a simple blending process, and this made it was selected for optimization [6]. On the other hand, PVA displayed some similar properties; PVA will interact and form a crosslinking network with PEDOT: PSS, and this interpenetrated structure displayed excellent mechanical flexibility [7]. Then, PEG can be used as a plasticizer when added to adjust the mechanical properties of PEDOT: PSS. As a stretchable film will yield after mixing PEDOT: PSS with PEG, the PEG in the film is used to prevent crack formation without influencing conductivity [8].

Thirdly, optimizing the acidity and stability of PEDOT: PSS is also an essential aspect. PSS component is highly acidic and will lead to corrosion of surrounding functional layers, and it will easily absorb moisture from the surrounding environment, which leads to phase separation of PEDOT and PSS, then reduces the conductivity and overall performance of the device. One approach to reduce acidity is the addition of a weak base such as imidazole or ammonium hydroxide. Those bases have weak redox properties; after they are doped into PEDOT: PSS, the styrenesulfonate group will be neutralized without reduction of conductivity [9]. To reduce the

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absorption of moisture and make PEDOT: PSS resistant to heat, some solutions are also proposed. One approach is the addition of perfluorinated ionomer (PFI). PFI will prevent the spreading of water molecules by forming a hydrophobic domain and suppress phase separation; thus, the stability of the PEDOT: PSS molecule will be improved [10]. At the same time, hydrophobic grafted side chains are also one approach. In this approach, a hydrophobic side chain will be grafted through the electro-polymerization process; the absorption of moisture is also reduced, and the structural and electronic stability is retained [11]. To make PEDOT: PSS have better robustness under heat, there are two aspects: crosslinking network and substitution of counter ions. For example, one crosslinking mechanism involves PVA, PSS, and (3-glycidyloxypropyl)trimethoxysilane (GOPS). After the hydroxyl group on PVA reacted with PSS, and GOPS bonded with PSS, a crosslinked network will be formed; the robustness and stability under heat improved [12]. On the other hand, substitution of counter ions also changed the structure and stability, making the PEDOT: PSS have better thermal stability.

2.3. Fabrication techniques

The fabrication of PEDOT: PSS devices involves several approaches, which can be selected based on desired morphology and target application.

The first aspect of implementation is coating. Because PEDOT: PSS can be dispersed into aqueous solution, it can be used in solution-based processes. This approach is cost-effective and compatible, as it offers relatively low cost, and more material can be utilized. Currently, several types of coating techniques have been developed, including dip coating, drop coating, spray coating, and spin coating. Dip coating can be used to form large areas of film on the surface of substrates, which is a special advantage in the fabrication of transparent flexible electrodes, but this approach is limited by the adhesive property of the material. In this process, a melamine sponge should be immersed in an aqueous dispersion of PEDOT: PSS to make sure dispersed PEDOT: PSS can be adhered to the micrometer-scale frameworks in the sponge. In comparison, drop coating is a much simpler process; in this step, PEDOT: PSS solution will be directly dropped onto the surface of the substrate without the support of special equipment. However, this approach also has disadvantages. Due to the evaporation of solvent, the thickness and size of the resulting films created by this process are difficult to manipulate, leading to poor uniformity. Like previous coating processes, the dispersion of PEDOT: PSS will be sprayed onto the surface of the substrate. This process can also be used for large-scale fabrication because a suspension of PEDOT: PSS can be sprayed onto large-scale substrates, such as the transparent flexible electrode. However, the main disadvantage of the spray coating technique is the relatively higher instrumentation requirements and associated equipment cost. The last coating process is spin coating. Compared with the methods above, spin coating offers high repeatability and uniformity on flat substrates. However, this approach has a disadvantage because it is not suitable for large-area fabrication and leads to significant material waste [13].

In addition to coating techniques, printing techniques are also a strategy for applying PEDOT: PSS to a substrate surface. This is a straightforward strategy, as PEDOT: PSS can be deposited on diversified types of substrates, and it has several advantages, including low cost, high efficiency, and less material waste. Currently, printing processes include three types, which are screen printing, inkjet printing, and 3D printing. For the first type of printing technique, screen printing is a mature process that can be used for various types of substrates; in this process, an aqueous mixture of PEDOT: PSS and poly (dimethyl siloxane-b-ethylene oxide) is screen-printed on knitted cotton to prepare conductive textiles. This method has some disadvantages because the film applied by this

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process is very thick, and this method is not appropriate for solutions with low viscosity and high volatility. In comparison, the second type of printing technique, inkjet printing, has displayed some benefits. This process can produce film with adjustable thickness, and it is simple and cost for it is relatively low, which is a better approach compared to the previous method that can only apply thick films. However, the charged ink droplets will affect the resolution and performance of the device. To address this issue, a third type of technique, which is 3D printing technique, has emerged. This technique can be used to rapidly print PEDOT: PSS film with arbitrary structure. Benefiting from its freedom regarding design, it has the capability to print structures with high complexity, and it has compatibility with a variety of materials. However, this method is hard to be used for printing complex and curved surfaces with high precision requirement, and its mechanical properties will be affected by the anisotropy of the product [13].

The third aspect of fabrication technique is conventional lithography, and it is also called top-down lithography. This is a widely implemented technique that is used for making micro or nanoscale structures on flat surfaces. One example of conventional lithography is the process of making microchips and integrated circuits, which made a great contribution to the semiconductor industry. There are three main types of conventional lithography, which are ordinary lithography, ion-beam lithography, and electron-beam lithography [13]. Ordinary lithography is the simplest approach; for example, a one-step photopatterning method based on photolithography has been proposed. In this process, using ultraviolet rays to transfer microstructures from a polydimethylsiloxane (PDMS) photomask to a PEDOT: PSS/polyethylene glycol hybrid film, thereby producing a microstructure thin film with a resolution of 2 μm [13]. In comparison, the other type of lithography, ion-beam lithography relies on the collision of an ion beam with high momentum. When high-momentum ion beams collide with the resist layer, resist particles will be removed. The high momentum means that ions propagate in a straight line, resulting in high-resolution etching. However, a high-momentum ion beam will also destroy substrates and reduce yield [13]. The third type of lithography is electron-beam lithography. Electron-beam lithography has extremely high resolution (below 10nm) owing to the relatively small wavelength of the electron beam. However, electron-beam lithography is time-consuming, and the process is complicated, which increases cost and makes this process only suitable for small-scale production processes [13]. Based on a comparison between conventional lithography techniques, each type of technique has its unique advantages; ordinary lithography is the simplest, which only relies on UV rays, and could be used for basic large-scale production processes. The other two types of conventional lithography are more accurate, but still have disadvantages such as high cost or low yield.

To provide an alternative approach to conventional lithography, which means some approaches should have properties such as low cost, high output, and being able to replicate micro- to nano-level structures on a large scale. Based on those expectations, alternative lithography has become an accessible commercial process that has been developed to resolve the limitations of conventional lithography. As an example of alternative lithography, soft lithography is a typical technique. In this process, elastomer impressions are used for printing, molding, transferring, and embossing. The result of this lithography relies on the contact between the elastomer and substrate. This approach has been applied to the fabrication of microfluidics, microelectronics systems, and flexible electronics/photonics. However, owing to the mechanical instability of the elastomer, this type of technique is usually limited to patterning micrometer-scale structures. The other type of alternative lithography is nanoimprint lithography. This is another commonly used lithography technique and can achieve high-resolution nanoscale patterning. Conventionally, this technique can be classified

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into thermal and ultraviolet imprinting. Hot embossing was the first stage of this technique, and it just requires a simple one-step heating and pressing process to transfer a pattern from a hard mold to a thermoplastic. During this process, the structure on the mold directly contacts the substrate in a molten state, and the "opposite" structure is replicated on the substrate. Although this technique can be used for creating very small structures (down to 5 nm), the high temperature or UV processes cannot fulfill the requirements of PEDOT patterning. To solve this problem, a hybrid nanoscale approach that combines the advantages of soft lithography and nanoimprinting has been developed [13].

2.4. Implementation

Because this polymer has several advantages, including good conductivity, high optical transmittance, superior flexibility, high uniformity, and low roughness, PEDOT: PSS became one of the representatives for FTE. Important aspects for the implementation of PEDOT: PSS in OLED include several aspects, which are hole transporting/injection layers, thermoelectric layers, perovskite hybrid components, motion- and temperature sensors, and stretchable conductors. For example, in recent studies, a report mentioned using a facile method for controlling the optoelectrical properties. In this approach, highly conductive and hole-injecting PEDOT: PSS solutions can be made by mixing PEDOT and PSS in different ratios. This mixture should undergo an annealing process at 200 degrees Celsius; after this process, the phase transition of PSS chains from PSS-rich domains initiates thermally induced phase separation. After this morphological change, the performance of FTE can be modified through different ratios [14].

3. Metal-based electrodes

Alternatively, besides the previously mentioned approach, there is another approach, which is metal based electrodes approach. In this approach, the low sheet resistance of metallic material will be implemented.

3.1. Basic properties and advantages

Currently, the metal-based transparent electrode focuses on three main categories: metal grids, metal nanowires, and ultra-thin metal films. In metallic film approaches, an ultra-thin film made from a noble metal will be implemented. This ultrathin film possesses relatively low sheet resistance and good mechanical robustness due to its inherited properties from bulk metal. In addition, compared with a thin metal film, metallic nanowire (NW) networks always show better transmittance and flexibility. For example, AgNWs based nano wire has been widely explored, and low sheet resistance (approximately $20\Omega^{-2}$) and high transmittance (approximately 80% in the visible spectrum region) have been found. Essentially, this nanofiber also has high transmittance in the near-infrared region and a comparable difference between diffusive transmittance and specular transmittance due to the light scattering effect; this property reflects the excellent performance of AgNWs-based nanofiber [14]. For transparent electrodes, this metallic nanofiber could be successfully implemented and considered as an appropriate substitute for ITO electrodes. For example, a solution-processed method to prepare the AgNWs-based FTE has been proposed and implemented and exhibited excellent optoelectrical properties (transmittance of 95% at 550nm and sheet resistance of $10\Omega^{-2}$) [14].

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3.2. Limitations with nano fibers

It should be noted that the limitations discussed below primarily pertain to metal nanowire (NW) networks and ultrathin metal films, as these are the two most widely investigated forms of FTEs. Metal grids, while circumventing the random junction issue, face separate challenges such as high fabrication costs and pattern resolution constraints, which are beyond the scope of this section. For AgNW-based networks in particular, the key bottlenecks are as follows. For nanofibers, roughness with surface texture is one essential restriction, and to deal with this restriction, researchers have devoted efforts. On the other hand, the non-uniform size of NWs will lead to random distribution on the substrate surface and influence the surface uniformity of the whole electrode, which would also deteriorate the subsequent film distribution and reduce the reproducibility of the device. Moreover, the contacts between metal NWs at cross junctions are weak, which might increase resistance at the junction and even increase the resistance of the resultant electrode. On the other hand, the properties of NWs are sensitive. For example, with increasing thickness, the conductivity will significantly increase at the same time, even approach to bulk metal, but the transmittance would sharply drop. To balance the transmittance and conductivity, the thickness of the metal film should be around 10nm, but the Volmer-Weber growth mode of metal at such a thin film is likely to form an island-like morphology, which usually exhibits low transmittance and electrical conductivity. Apart from the previous properties, excellent adhesion between the electrode and substrate should also be considered, because poor electronic contact would greatly deteriorate the relative device performance [14].

3.3. Possible Solutions

To address previous issues, there are several strategies. Firstly, considerable efforts have been devoted to reducing the surface roughness and enhancing the conductivity by precisely manipulating the size distribution of metal NWs and improving the contact between metal NWs at cross junctions. For example, Chen et al proposed an ionic electrostatic-charge-repulsion method by doping a polyelectrolyte into a silver NW solution to reduce the aggregation of AgNWs and the junction resistance. The resultant flexible electrode has a grid-like structure that could successfully decrease the sheet resistance to 10 Ω^{-2} has a high transmittance of around 92%. As a result, the resultant FTE can be more efficient, such as power conversion efficiency around 16.5% and better mechanical stability [15]. On the other hand, to address the sensitivity of FTEs, some solutions have also been proposed. For example, a process which used pre-deposited a zinc oxide layer to improve the growth of copper (Cu) on a flexible substrate and obtained a continuous and smooth ultrathin Cu film. This process made the film more sensitive, which is a great improvement. To address the third problem, the composite electrodes combining nanopatterned metal material with other semiconductive/conductive materials were explored to address the problem of electronic adhesion.

4. Challenge and prospects

4.1. Challenges

Currently, although both strategies have been recognized as possible solutions for future OLEDs, there are still challenges associated with them. Firstly, there are too many intrinsic limitations related to the PEDOT: PSS and silver-based nanofibers. Besides the intrinsic limitations of the previously mentioned materials, the structural design of FTE faced other problems. For example, the most

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widely implemented multilayer architecture is the Dielectric-Metal-Dielectric structure (DMD structure), which inserts an ultrathin metal layer between two oxide or polymer layers to improve both transmittance and conductivity of the electrode. Research on this structure has reached a considerable level of maturity, and DMD structures exhibit excellent electrooptical properties. However, besides the superior properties, the cost of a multiple-layer fabrication process involving repeated evaporation and deposition steps is very expensive. This process not only substantially raises manufacturing costs but also greatly makes the roll-to-roll scale production more complicated. Secondly, as discussed above, two approaches face inherent challenges that require sustained research efforts. When FTEs based on conductive polymers and metal-based nanowires are primarily implemented, their limitation become evident. Conductive polymers require long-term treatment to adjust their intrinsic property, while fabrication methods remain imperfect--they are either expensive, inaccurate, or both. And nanofibers still have similar conditions; to be further implemented, the FTE made from AgNWs should be more stable and less sensitive, and this nanowire structure is complicated to be produced in large scale.

4.2. Prospect

There are still many expectations for the development of FTE in the OLED field. Firstly, different fabrication techniques and production methods should be integrated; the integrated strategy should be less time-consuming, more cost-effective, and more accurate. Moreover, the development of FTE in the future should focus on better robustness in elongation scenarios, because current FTE mainly focuses on flexibility in folding scenarios. As a result, the FTE with intrinsic robustness under elongation should be developed in the future. To make sure the FTE can be produced on a larger scale, the roll-to-roll production method and other types of low-cost production methods, such as inkjet imprinting, should cooperate with each other to get a better implementation in the industry field. The long-term development should focus on composite FTE, because both polymer and metal-based materials have intrinsic disadvantages, but this defect will become less serious when a composite material is formed. A new type of material should be more accurate, which means it accurately corresponds to different emission energy levels, which means more implementation opportunities.

5. Conclusion

In conclusion, this research has yielded key findings in several aspects. To find a substitute for ITO electrodes, the strategy includes conductive polymer and metallic nanofibers. The conductive polymer strategy currently focuses on PEDOT: PSS, owing to its low roughness, high conductivity, high transmittance, and ease of fabrication. However, this polymer also has disadvantages, including low intrinsic conductivity, moisture absorption, and high acidity. To deal with these restrictions, some strategies to modify the intrinsic properties have been developed, and different fabrication techniques have been found, including printing and lithography. In the future, new optimization methods should be found, and different types of fabrication techniques should be integrated because previous research only relied on conventional strategies, and fabrication techniques are still imperfect. On the other hand, the other approach has similar conditions: as a type of sensitive material, nanofiber has inherited advantages from bulk metal, high transmittance, but it is too unstable and experiences difficulties as relatively high resistance at the junction because of complex distribution. Based on those issues, modification of the morphology of nanofiber is required to make it less complicated and more stable in the environment. Moreover, both strategies should be

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integrated to overcome the current limitations and develop new FTE structures beyond the current DMD structure. Finally, the FTE should be designed for stretchability, as currently the FTE is primarily optimized for folding conditions. These prospects point toward the need for integrated strategies that yield FTEs with both robust stretchability and stable optoelectronic properties.

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