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Thin film encapsulation for flexible AM-OLED: a review

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Abstract
Flexible organic light emitting diode (OLED) will be the ultimate display technology to customers and industries in the near future but the challenges are still being unveiled one by one. Thin-film encapsulation (TFE) technology is the most demanding requirement to prevent water and oxygen permeation into flexible OLED devices. As a polymer substrate does not offer the same barrier performance as glass, the TFE should be developed on both the bottom and top side of the device layers for sufficient lifetimes. This work provides a review of promising thin-film barrier technologies as well as the basic gas diffusion background. Topics include the significance of the device structure, permeation rate measurement, proposed permeation mechanism, and thin-film deposition technologies (Vitex system and atomic layer deposition (ALD)/molecular layer deposition (MLD)) for effective barrier films.

1. Introduction

With superior display quality such as vivid full color, perfect video capability and thin form factor, AMOLED (active matrix organic light-emitting diode) is making its headway into mobile devices, and OLED TV (television) is poised to enter into consumer markets in the near future [1, 2]. It is quite natural that AMOLED on flexible substrates will be deemed as the ultimate display because of its ruggedness and flexibility. However, there are a number of challenges to realizing flexible AMOLED, especially on plastic substrates, e.g., low-temperature thin-film transistors with good performance and high reliability, flexible electrodes, and thin-film encapsulation (TFE) [3–6]. Among these challenges, TFE is the most demanding task because OLED requires the highest degree of protection from moisture and oxygen penetration in electronic devices. An overview of the barrier requirements for various applications is given in figure 1 [7]. In fact, the well-known approach so far is to form multiple layers of organic and inorganic thin films, so called Vitex technology.

Five dyads of organic and inorganic combination have been demonstrated to have WVTR (water vapor transmission rate) under $10^{-6}$ g/m$^2$/day, which is adequate for OLED [8]. However, it requires high COO (cost of ownership) due to low throughput and high investment. Recently, multilayers of ALD (atomic layer deposition)/MLD (molecular layer deposition) combination have been proposed as an alternative to Vitex technology [9]. The main advantage of this approach is that it can achieve low WVTR with fewer layers because ALD/MLD layers have much better film integrity, but the drawback has been the low throughput and limitation in the scalability of the ALD/MLD process.

We will discuss the various considerations for the device structure and encapsulation techniques for flexible AMOLED. Barrier requirements and permeation barrier measurement techniques will be also discussed. Based on reviewing the degradation mechanism of OLED in moisture and oxygen environment, the state of the art in thin-film permeation barriers and multilayer structures, including Vitex technology and ALD process are discussed and the barrier properties are provided. Finally, we will discuss how to realize high throughput and scalable ALD/MLD.
2. Device structures for flexible OLEDs

There are various device structures possible for the fabrication of OLED displays. Figure 2(a) shows a schematic diagram of the conventional OLED device structure. The OLED structure consists of two or more organic layers in between a transparent anode (ITO: indium tin oxide) and a metal cathode [10]. As most OLED work has been focused on the development and manufacture of glass-based displays, encapsulation has been achieved by sealing the display in an inert atmosphere such as nitrogen or argon using a glass lid or metal can secured by a bead of UV-cured epoxy resin [11]. A getter such as calcium oxide or barium oxide is often incorporated into the OLED device to react with any byproducts of the resin cure process and any residual water incorporated in the device or diffusion through the epoxy seal.

On the other hand, typical encapsulation techniques have some problems to apply flexible OLED displays [5, 6, 12] due to the rigidity of the substrate and the lid. Recently, various flexible encapsulation methods have been developed, including barrier-coated flexible lids [7, 13] and thin-film barrier coatings [14–16] in intimate contact with the display surface. Figure 2(b) exhibits the representative scheme of the TFE structure. The advantages of the TFE method with a thinner/lighter form factor and a higher flexibility of device form during in-flex use of the display. However, there are still several issues to adopt the TFE methods, i.e. the process must be performed at relatively low temperatures and any contact to the OLED by adverse agents, such as solvents, must be minimized to prevent damage to the OLED components.

The most important candidates in substrate materials for flexible AMOLEDs are polymers [17], metal foils, and ultrathin glass. Thin glass and metal foils are both almost similar to the rigid glass substrate as the effective barrier properties, needing no more barrier layers on the flexible substrate (but a flexible top barrier is still required). Polymer substrates provide better flexibility than glass or metal and better toughness (not brittleness) than glass, but have insufficient barrier properties to water and oxygen permeation. According to several reports [18, 19], the water and oxygen permeation rates for several polymer substrates are around $10^{-1} \sim 40 \text{ g/m}^2\text{/day}$ and $10^{-2} \sim 10^2 \text{ cm}^3\text{/m}^2\text{/day}$, respectively, depending on molecular weight, density and polarity of monomers. Therefore, an additional barrier layer on the polymer substrate is required. In particular, one of the most important considerations for permeation barrier is surface quality. The thickness of the OLED layer is typically of the order of 100–200 nm and a large electrical field is applied during its operation. Thus, non-uniformity in the anode film can induce in local regions of very high field, leading to deleterious effects of the OLEDs performances, such as a short-circuited pixel or the formation of dark spots and device degradation [20]. Unlike glass, polymer substrates cannot be smoothed by mechanical surface polishing. The roughness of the surface is particularly problematic [21, 22], causing the efficiency and lifetime of OLED devices to deteriorate. In terms of flexible polymer substrates, the smooth diffusion barrier films on the polymer should be required due to high permeability and surface roughness.

3. Measurement of permeation rates

Permeation of oxygen and water vapor was measured with standard devices from Mocon [23] and Brugger [17]. Both types have a measurement range starting at $0.005 \text{ cm}^3\text{/m}^2\text{/day}$ or $0.005 \text{ g/m}^2\text{/day}$ respectively. The permeated species are detected by a coulometric sensor or electrolysis, respectively. However, none of the commercially available systems based on these techniques meet the sensitivity requirements for the low permeation rates required for OLEDs. As mentioned in figure 1, the WVTR should be measured reliably at least as low as $10^{-6} \text{ g/m}^2\text{/day}$ to develop sufficient barriers for OLEDs. Therefore, new measurements have been developed.

One approach is known as the Ca test, showing a schematic diagram of the Ca test package in figure 3(a). Ca is a reactive material with water and oxygen and also a conductive metal, which has a resistance against an applied voltage [24, 25]. This involves observation of the optical or electrical
changes as an opaque reactive metal, such as Ca, that converts to a transparent oxide insulator or hydroxide salt [26, 27]. The optical Ca test measures the transmission of O specie since the Ca is oxidized to either CaO or Ca(OH)$_2$ [28, 29]. On the other hand, the resistance Ca test allows determining the degree of Ca corrosion with high accuracy, measuring that initial height of the Ca layer was normalized by an initial current under applying a constant voltage. Reported effective transmission rates for H$_2$O through barrier films using the Ca test are as low as 3 × 10$^{-7}$ g/m$^2$/day [29]. The Ca test has the advantage of discriminating between bulk permeation and defect-based permeation, which can be observed as spots on the Ca film. However, it does not discriminate between oxygen and water permeation. This suggests that the sensitivity for WVTR is an upper limit since other permeants could have been present. Permeation techniques using an ultra-high vacuum have been used to obtain OTR (oxygen transmission rate) measurements as low as 1 × 10$^{-6}$ cm$^3$/m$^2$/day by a residual gas analyzer [18]. But the same techniques for water are very difficult due to the longer pumping times.

To measure reliable water permeation, the other approach is known as the tritium transmission rate (TTR) measurement, using the radioactive HTO (hydrogen-tritium-oxygen) tracer method [30–32]. HTO is the source of tritium that can diffuse through the films either as molecular HTO or possibly as tritium atoms. Figure 3(b) exhibits the schematic diagram of TTR measurement. The LiCl absorbed the tritium contained in the HTO that had permeated through the polymer film. The LiCl also absorbed residual H$_2$O from the walls of the test cell or H$_2$O from the outside atmosphere. The scintillation counter used to count the tritium decays of LiCl-containing HTO and to calculate the HTO transmission rates. The detection limit of WVTR is below ∼1 × 10$^{-6}$ cm$^3$/m$^2$/day.

### 4. Degradation mechanisms of vapor permeation

Flexible OLED test pixels were first demonstrated by Gustaffson et al [12] using polymeric electroluminescent layers in 1993, and later by Gu et al [33] in 1997 using small molecules (nonpolymeric) organic emitters. Despite the potential advantages of an extremely thin, flexible, emissive display, the commercialization of flexible OLEDs is hindered by their extreme sensitivity to moisture. In particular, major degradation mechanisms have been suggested due to metal delamination, recrystallization of organic materials, reactions in metal and organic interfaces, metal migration, molecule-specific degradation, oxidation by water and oxygen, and so on [11, 34–36]. Among these, water and oxygen is known to cause significant degradation of OLED devices. In the presence of oxygen and water, almost all pi-conjugated molecules and polymers undergo oxidation during the device operation. Degradation by water is more acute than by dry O$_2$ [37]. Thus, the required WVTR is less than 10$^{-6}$ g/m$^2$/day [38, 39].

Recently, Graft et al [40] have reported time-dependent permeation data to calculate the effective diffusivity ($D$) and solubility ($S$) for water vapor through an aluminum oxide (AlO$_x$) barrier layer used in a multilayer ultra-barrier stack as shown in figures 4(a) and (b). They suggest that the effective diffusivity and solubility of water vapor in such vacuum-deposited AlO$_x$ layer is orders of magnitude higher than that of pristine, fully dense crystalline alumina, indicating the dominance of defects on vapor permeation. Although the equilibrium permeability is theoretically equal to the product $DS$, there are still considering factors (such as diffusion lag time, grain boundary, spatial density, defect size etc [22, 40, 41]) to estimate the practical permeability. This effective $D$ had been evaluated by an equivalent defect size at a defect spacing, and table 1 summarized the measured defect sizes and defect densities for single-layer inorganic barrier films deposited on polymeric substrates. Therefore, the
Figure 5. A cross-sectional scanning electron microscopy (SEM) of the multilayer barrier layer (inorganic/organic layers): four pairs of AlO\x/x/polymer multilayer deposited by the Vitex barrier deposition system.

Table 1. Summary of the measured defect size and defect densities for single-layer inorganic barrier films deposited on polymeric substrates.

<table>
<thead>
<tr>
<th>Defect radius (nm)</th>
<th>Defect density (mm(^{-2}))</th>
<th>Coating material</th>
<th>Deposition method</th>
<th>Substrate</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.6</td>
<td>11–1100 SiO(_2)</td>
<td>PECVD</td>
<td>PET</td>
<td>[41]</td>
<td></td>
</tr>
<tr>
<td>0.6</td>
<td>5–1000 Si(_3)N(_4)</td>
<td>PECVD</td>
<td>PET</td>
<td>[41]</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>25–400 Al</td>
<td>Evaporated</td>
<td>PET</td>
<td>[40]</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>100–300 Al</td>
<td>Evaporated</td>
<td>PET</td>
<td>[22]</td>
<td></td>
</tr>
<tr>
<td>2–3</td>
<td>200</td>
<td>Sputtering</td>
<td>PET</td>
<td>[42]</td>
<td></td>
</tr>
<tr>
<td>0.5–1.4</td>
<td>600</td>
<td>AlO(_3)N(_y)</td>
<td>Sputtering</td>
<td>PET</td>
<td>[43]</td>
</tr>
<tr>
<td>0.4</td>
<td>100–1000 Al</td>
<td>Evaporated</td>
<td>BOPP</td>
<td>[44]</td>
<td></td>
</tr>
<tr>
<td>0.5</td>
<td>700</td>
<td>AlO(_3)N(_y)</td>
<td>Sputtering</td>
<td>PET</td>
<td>[45]</td>
</tr>
</tbody>
</table>

measured properties can be used to predict the permeability of gases in single- and multilayer thin film barrier stacks.

In addition, this research allows us to propose design criteria for ultra-barrier films for flexible OLEDs: (1) permeation through barrier films is controlled by defects; defect size and spatial density in the films are the critical parameters. (2) The long apparent diffusion path induced by sandwiching thin polymer layers between low defect density inorganic layers causes a pronounced increase in lag times but a much less significant decrease in the steady-state flux. (3) Lowering the diffusivity and solubility of the polymer interlayers will improve barrier performance.

5. Vitex technology

Various single-layer materials have been investigated for barriers. The high density of barrier films, including silicon nitride, Al, Ta, and Si oxide, results in better barrier performances for these materials than for defective and porous films [46–48]. Although numerous materials have also been evaluated using sputtering, evaporation, and plasma vapor deposition, the performance improvement was minimal [49–51]. Only a single-layer barrier structure is short of the requirement of flexible OLEDs, but significant improvement in permeation–barrier performance can be achieved by using multilayer structures [52, 53].

Nowadays, alternating inorganic and organic layer pairs are suggested as an encapsulation solution for flexible devices. Vitex System is one of the pioneers in the field of flexible encapsulation and they reported flexible encapsulation layers (trade name of Barix®) made of alternating Al\(_2\)O\(_3\) and polycrylate layers as shown in figure 5 [16, 54, 55]. The Al\(_2\)O\(_3\) layers are reactively sputtered onto the display via an energetic plasma between polycrylate layers, which are deposited via flash evaporation of the monomer followed by ultraviolet (UV) curing [54]. A polycrylate layer has been attributed to smoothing of the substrate, reduced mechanical damage, increased thermal stability of the nucleation surface, and increased chemical polarization [56, 57]. Inorganic Al\(_2\)O\(_3\) layers are working as thin barrier layers. By repeating the alternating process to deposit multiple layers, the polymer films decouple any defects in the oxide layers, thereby preventing propagation of defects through the multilayer structure. When the organic layer thickness is less than the average size of pinholes (defects) in the inorganic barrier layer (Al\(_2\)O\(_3\)), the permeation process is governed by a ‘tortuous path’ mechanism with a strong dependence of the WVTR on the organic layer thickness. Thus, the multilayer structure may provide longer diffusion paths of permeants and lower pinhole densities to improve the barrier performance [57, 58].

With this general approach Vitex and others claim WVTRs approaching the 10\(^{-6}\) g/m\(^2\)/day target. As shown in figure 5, the thickness of the inorganic layers is in the order of tens of nanometers and polymers are in the micrometer range. The total thickness of the layers is a few \(\mu\)m. They achieved half-luminescence lifetime of about 2500 h which is a quarter of the performance of glass encapsulated devices.

On the other hand, the WVTR was determined with the calcium test as shown in figure 6. Water permeation was significantly reduced with three pairs of inorganic and inorganic layer pairs. Performance with passive matrix OLED was also demonstrated as shown in figure 7 [16].

Even though the encapsulation solutions of inorganic and organic layer pairs show excellent performance for flexible devices, their manufacturability is not proven at this moment. The inorganic layer requires high cost vacuum equipment and its production throughput is very low. Various approaches are being made by many researchers. Most of the barrier layers have an inorganic and organic multilayer structure to achieve low water permeability and high flexibility. To overcome the low throughput issues, a continuous process was suggested.
be significantly enhanced. However, further research and development is required for the commercialization of the continuous process concept.

6. Atomic layer deposition/molecular layer deposition technology

ALD had been developed and introduced worldwide with the name atomic layer epitaxy (ALE) in the late 1970s [60–62]. The first application of ALE was thin-film electroluminescent displays, requiring high-quality dielectric and luminescent films on large area substrates [63]. And then, interest in ALD has increased with focusing on silicon-based microelectronics. ALD is considered as one deposition method with the greatest potential for producing very thin, conformal films with control of the thickness and composition of the films possible at the atomic level [64, 65]. These promising potentials have recently led to scaled down microelectronic devices. ALD can be used to deposit several types of thin films, including various oxide, metal nitrides, metals, metal sulfides [66–69]. Also, other applications requiring continuous and pinhole-free outside of the semiconductor industry are the diffusion barrier coatings with low gas permeability [22] and low electron leakage dielectrics for organic photovoltaic cells [70].

ALD is a sequential, self-limiting surface reaction that deposits conformal thin films of materials onto the substrate of varying compositions. A schematic showing the sequential, self-limiting surface reactions during ALD is displayed in figure 8. Most ALD processes are based on binary reaction sequences where two surface reactions occur and deposit a binary compound film. Because there are only a finite number of reactant ‘A’ chemisorption sites, the reactions can only deposit a finite number of reactant ‘B’ molecules [71]. Basically, ALD is similar in chemistry to chemical vapor deposition (CVD), except that the ALD reaction breaks the CVD reaction into two half-reactions, keeping the precursor materials separate during the reaction, so called ‘purging’. Separation of the precursors is accomplished by pulsing a purge gas (typically nitrogen or argon) after each reactant pulse to remove excess precursor from the process chamber and prevent parasitic CVD deposition on the substrate. If each of the two surface reactions is self-limiting, then the two reactions may proceed in a sequential fashion to deposit a thin film with atomic level control.

The advantages of ALD are the precise thickness control on the number of reaction cycles. Unlike CVD, there is less need of reaction flux homogeneity, providing large area uniformity, excellent conformality on the high aspect ratio structure and reproducibility [72]. In addition, the self-limiting aspect of ALD produces extremely smooth, very continuous and pinhole-free film because no surface sites are left behind during film growth. This factor is extremely important for the TFE techniques to improve diffusion barrier properties.

On the other hand, similar self-limiting surface reactions can be employed for the growth of organic polymers. This film growth is described as MLD because a molecular fragment is deposited during each ALD cycle [73]. MLD was initially developed for the growth of organic polymers [59]. In this concept a continuous roll-to-roll process in vacuum was suggested. In this way process throughput can
such as polyamides [74] and polyimides [73]. Nowadays, a number of other organic polymers, including poly(p-phenylene terephthalamide) (PPTA) [75], polyurea [76], polyurethane [77] and polyazomethine [78], have also been grown using MLD techniques.

Among emerging applications, the low temperature ALD enables us to create unique inorganic/organic polymer composites and to deposit thin and conformal diffusion barrier films on thermally sensitive materials such as organic polymers. In particular, Al2O3 ALD has been employed to passivate or encapsulate OLED and organic solar cells to prevent H2O permeation [70, 79–82]. Many researchers have demonstrated the gas diffusion barrier films on polymer as summarized in table 2 [79–85]. At the beginning, plasma-enhanced atomic layer deposition (PEALD), enabling deposition at low temperature due to high energetic radicals, has been used to deposit the Al2O3 thin film below 100 °C [79]. However, the barrier films by PEALD exhibit poor WVTR and insufficient OLED lifetime after encapsulating the device. Recently, George’s Groups [9, 80, 86] have reported excellent barrier properties for polymeric substrates coated with a thin layer of Al2O3 ALD, showing that thin films of Al2O3 ALD on polymeric substrates reduced the WVTR from ~1 to <10−5 g/m2/day at room temperature. The ALD diffusion barrier films are very attractive as TFE layers to meet the demanding needs of highly sensitive OLEDs for emerging flexible display and lighting technology. However, the major limitation of ALD is its slow growth rate (low throughput). Usually, only a fraction of a monolayer is deposited in one cycle. Therefore, the researchers have developed the multilayer structure with the goal of preventing the H2O permeation and improving the ALD coating time.

At this time, the MLD of hybrid organic–inorganic polymers has been demonstrated using inorganic precursors from ALD with various organic precursors [87]. One class of the hybrid-inorganic MLD polymer is the alucones [88]. These hybrid polymers result from using aluminum alkyl precursor such as trimethylaluminum (TMA) and various organic diols such as ethylene glycol (EG) [88]. Figure 9 shows the multilayer structure with Al2O3 and alucone layers, deposited by using TMA, EG and water. In fact, the MLD of alucones is very robust and yields very efficient and linear growth but the MLD process is still developing to solve the issues such as thermally sensitive organic precursors and naturally porous organic films [87].

Even though the ALD deposition is possible to deposit on a large area substrate, in terms of a TFE process, ALD and/or MLD processes should satisfy as low cost and high throughput processes. The cost of ALD is largely tied to the cost of the reactants and the equipment. Most ALD is performed with

**Figure 8.** Schematic representation of ALD using self-limiting surface chemistry and an AB binary reaction sequence.

**Table 2.** Summary of the barrier properties of ALD thin films including material, deposition condition and barrier structure. (N/D: no data.)

<table>
<thead>
<tr>
<th>Process</th>
<th>Materials</th>
<th>Deposition condition</th>
<th>Barrier layer structure</th>
<th>WVTR (g/m²/day)</th>
<th>OLED lifetime</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEALD</td>
<td>Al2O3:N</td>
<td>TMA, O2, N2</td>
<td>300 nm thick</td>
<td>N/D</td>
<td>650 h</td>
<td>79</td>
</tr>
<tr>
<td>ALD</td>
<td>Al2O3</td>
<td>TMA, H2O</td>
<td>25 nm thick</td>
<td>1.7 × 10−5</td>
<td>N/D</td>
<td>80</td>
</tr>
<tr>
<td>ALD</td>
<td>Al2O3</td>
<td>TMA, H2O</td>
<td>30 nm thick</td>
<td>0.0615</td>
<td>193 h</td>
<td>81</td>
</tr>
<tr>
<td>PEALD</td>
<td>Al2O3</td>
<td>TMA, O2</td>
<td>10—40 nm thick</td>
<td>5 × 10−3</td>
<td>N/D</td>
<td>82</td>
</tr>
<tr>
<td>PEALD</td>
<td>TiO2</td>
<td>TDMAT, O2</td>
<td>80 nm thick</td>
<td>0.024</td>
<td>90 h</td>
<td>83</td>
</tr>
<tr>
<td>ALD</td>
<td>Al2O3/SiO2</td>
<td>TMA, Silanol, H2O</td>
<td>5 nm (Al2O3)/60 nm (SiO2) bilayer</td>
<td>5 × 10−5</td>
<td>N/D</td>
<td>9</td>
</tr>
<tr>
<td>ALD</td>
<td>Al2O3/ZrO2</td>
<td>TMA, TDMAZr, H2O</td>
<td>2.1 nm (Al2O3)/3.1 nm (ZrO2) total 100 nm thick</td>
<td>4.75 × 10−5</td>
<td>10000 h</td>
<td>84–85</td>
</tr>
</tbody>
</table>
Figure 9. A cross-sectional scanning electron microscopy (SEM) of the multilayer layers (Al2O3/Alucene) deposited on the polymer substrate by ALD and MLD processes.

Figure 10. A schematic diagram of the atmospheric pressure ALD system.

vacuum pumps that act to move the reactants and products through the reactor and maintain a clean environment in the reactor. To achieve the issue, if higher pressure than the atmospheric pressure pushes the reactant and product gases in the reactor, the atmospheric pressure ALD will be possible without vacuum pumps. Recently, a few groups have developed atmospheric pressure ALD based on a coating head that is positioned onto a substrate as shown in figure 10 [89, 90]. The ALD reactants are delivered through channels in the coating head. The channels are separated by inert gas flow to prevent the gas phase reactions of the precursors. The coating head could either move in a linear fashion or move back and forth to overlap the surface areas and achieve ALD processes as shown in figure 10. Thus, the novel ALD system in the TFE techniques will provide a solution to overcome the limitation of cost and throughput in large area substrates.

7. Summary

OLED technologies are persistently facing the demands of markets and customers. The realization of flexible OLED applications still requires further advances in the technology of TFE. Several barrier architectures are possible and each technology has different materials, processing and barrier properties. Moreover, the measurement of permeation rate must be improved for developing the technology of TFE. The mechanisms of gas permeations have been proposed and investigated with various experiment and models, which help to understand the considering factors of designing barrier films. Thus, better understanding of the synergistic effect of polymer/inorganic multilayers (Vitex Technology) may lead to improved barrier performance from simple structures but there are still issues to manufacture practically the TFE on large area/flexible OLED application. Recently, the ALD and/or MLD technologies may give clues to solve the issues of TFE. Therefore, a novel ALD and/or MLD system (Synos Technology) will provide a big breakthrough and opportunity of TFE in OLED industry. This review shows present issues to consider large area/flexible OLED applications and will help to realize a truly next generation AMOLED technology.

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