Thin-Film Permeation-Barrier Technology for Flexible Organic Light-Emitting Devices

Jay S. Lewis and Michael S. Weaver

Invited Paper

Abstract—One of the advantages of organic light-emitting devices (OLEDs) over other display technologies is the ability to fabricate them on flexible substrates. As polymer substrates do not offer the same barrier performance as glass, OLEDs on polymer substrates will require thin-film barriers on both the bottom and top side of the device layers for sufficient lifetimes. This article provides a review of permeation-barrier technologies as well as the current status of thin-film permeation barriers for OLEDs. Topics include the implications of various device structures, permeation rate measurement, background and state-of-the-art of barrier technology, and mechanical and optical considerations for effective barriers.

Index Terms—Flexible displays, organic light-emitting devices (OLEDs), permeation barriers.

I. INTRODUCTION

RAPID progress in the performance of organic light-emitting devices (OLEDs) has positioned the technology as a potential replacement for many liquid crystal display applications. While the convention in the field is to refer to small molecule-based devices as OLEDs and polymer-based light-emitting devices as PLEDs, the role of the permeation barrier is similar. Therefore, for the purposes of this review, the term OLEDs will refer to both technologies inclusively. One advantage of OLEDs, because of the efficient, emissive, solid-state nature of the devices and the absence of viewing-angle effects, is the ability to fabricate them on a flexible substrate [1], [2]. Fabrication of active-matrix backplanes on polymer substrates is a challenge, but several groups have demonstrated both passive- and active-matrix displays on polymer substrates [3]–[7]. However, the hurdle for the introduction of flexible OLEDs (FOLEDs) into commercial applications is the limited lifetime exhibited by these displays. Effective encapsulation to prevent the ingression of water and oxygen to the device layers is required to achieve adequate lifetimes. This is already a challenge on rigid substrates such as glass, which provides an effective barrier on one side of the device [8]. For flexible substrates, thin film barriers are used in place of the rigid glass or metal to effectively isolate the device from exposure to moisture or oxygen. The various considerations for device structures and encapsulation techniques for FOLEDs will be presented. Barrier requirements and permeation barrier measurement techniques will also be discussed. Much of the available literature regarding thin film barriers involves metal or metal-oxide films on polymer substrates for food or pharmaceutical packaging. That research provides insight into the challenges that lie ahead for OLED permeation barriers, so an overview is given for that technology as well as for alternative materials and processes. The state of the art in thin-film permeation barriers and multilayer structures for OLEDs are discussed and device lifetime results are provided. Finally, the brittle nature of effective thin film barriers and the mechanical behavior of these films on flexible substrates will be discussed, as will the potential optical effects of transparent multilayer films on displays.

II. DEVICE STRUCTURES FOR FLEXIBLE OLEDs

There are numerous device architectures possible for the fabrication of OLED displays. The choice of device architecture impacts the requirements for barriers, and several options will be introduced here briefly. A basic OLED structure consists of two or more organic layers in between a transparent anode and a metal cathode [9]. The device structure is completed with some form of encapsulation above the cathode. If the substrate is permeable, as in the case of most flexible plastic materials, then an additional permeation barrier beneath the device layers is also necessary.

When a forward bias voltage is applied across the electrodes, holes and electrons are injected at the anode and cathode, respectively, and move through the device under the influence of the applied electrical field. These charges can then combine, forming excited molecular species, e.g., excitons, some of which may then emit light as they decay to their ground state.

A. Encapsulation Methods

As most OLED work to date 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 [10]. A getter such as calcium oxide or barium oxide is often incorporated into the package to react with any byproducts of the resin cure process

J. S. Lewis is with the MCNC Research and Development Institute, Research Triangle Park, NC 27709 USA (e-mail: jlewis@mcnc.org).

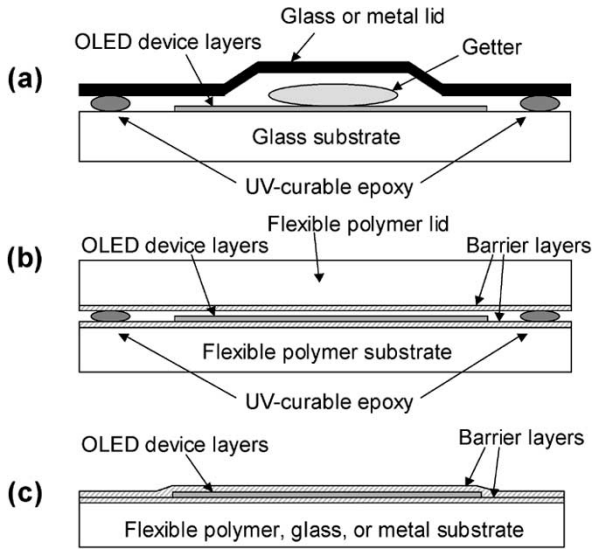M. S. Weaver is with the Universal Display Corporation, Ewing, NJ 08618 USA (e-mail: mikeweaver@universaldisplay.com).

Fig. 1. Schematic diagrams for OLED encapsulation structures: (a) traditional; (b) laminated barrier-coated lid; and (c) monolithic thin film.

TABLE I
WVTR AND OTR FOR VARIOUS POLYMERS AND COATINGS [19], [20], [47], [48]

POLYMER	$WVTR^a$ (g/m^2/day) (37.8-40°C)	OTR^c (cm^3 (STP)/m^2/day) (20-23°C)
Polyethylene	1.2-5.9	70-550
Polypropylene (PP)	1.5-5.9	93-300
Polystyrene (PS)	7.9-40	200-540
Poly(ethylene terephthalate) (PET)	3.9-17	1.8-7.7
Poly(ethersulfone) (PES)	14^b	0.04^b
PEN	7.3^b	3.0^b
Polyimide	0.4-21	0.04-17
15nm Al/ PET	0.18	0.2-2.9
SiO$_x$/PET		0.007-0.03
ORMOCER/PET		0.07
OLED requirement (estimate)	1 x 10^{-6}	1 x 10^{-5} - 1 x 10^{-3}

^{a} Calculated assuming 100μm polymer film

^{b} Temperature not given

^{c} Calculated assuming a 100μm polymer film and 0.2 atm O$_2$ pressure gradient

and any residual water incorporated in the package or diffusing through the epoxy seal over time. A schematic diagram of this structure is shown in Fig. 1(a).

For FOLED displays [1], [11], [12], conventional encapsulation techniques are ineffective due to the rigidity of the lid. Currently, several flexible encapsulation approaches are being developed. These include barrier-coated flexible lids [13], [14] and thin-film barrier coatings in intimate contact with the display surface [15], [16], shown schematically in Fig. 1(b) and (c). The advantages of the thin-film direct-encapsulation approach are a thinner form factor and a lesser concern for abrasion damage from the lid during in-flex use of the display (although the thin film barrier itself must be robust enough for handling by the user). However, the thin-film barrier deposition process must be compatible with the OLED beneath, 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 active components. The barrier coated lid approach has the advantage of allowing the manufacturer greater flexibility in terms of process conditions as the barrier is deposited onto a relatively robust surface, i.e., the lid material, as opposed to the more fragile OLED display. Typically, the lowest-permeability epoxies are rigid, so effectively sealing the laminated lid could be a challenge [17]. Mechanical considerations that differentiate these structures will be discussed in Section VI.

While the encapsulation approaches illustrated in Fig. 1 are the most often mentioned, there are other options as well. For example, if an impermeable substrate is used such as ultrathin glass or metal foil [18], substrate barrier layers are not required. If two high Young's modulus materials are used, such as glass/glass or foil/glass, then a curved display can be achieved and permeation barriers are unnecessary. However, these curved structures will not have the flexibility of a polymer-based structure. In the case of a high modulus substrate, either monolithic thin films or laminated top barriers may be used. However, the mechanical considerations for a

thin film barrier on a polymer versus high-modulus substrate are very different.

B. Substrates

The leading candidates in substrate materials for FOLEDs are polymers, metal foils, and ultrathin glass. Thin glass and metal foils both provide the same effective barrier properties as rigid glass substrates, needing no further barrier layers. A flexible top barrier is still required. Polymer substrates provide better flexibility compared to metal or glass and better ruggedness than glass, but do not provide sufficient protection to water and oxygen permeation. This can be seen in the permeation rates for several common substrate materials given in Table I together with the requirements for OLEDs. (A more extensive review of permeation rates can be found in [19] and [20].) Therefore, a substrate barrier structure is required in addition to a top barrier.

One of the most important considerations for substrates regarding permeation barriers is surface quality. The thickness of the organic layers in an OLED is typically of the order of 100 to 200 nm and a large electrical field is applied during its operation (of the order of 10^6 V/cm). Therefore, any appreciable nonuniformities such as "spikes" in the anode film can result in local regions of very high field which can, in turn, lead to deleterious effects on the OLEDs performance, such as a short-circuited pixel, or the formation of dark spots and device degradation [21], [22]. Unlike glass, polymer substrates cannot be polished and surface roughness is particularly problematic [3], [23]. For example, commercially available heat-stablized polyethylene terephthalate (PET) substrates [24] can have surface asperities greater than 150 nm. Therefore, any barrier structures deposited onto the substrate polymer before deposition of the OLED device layers must provide an exceptionally smooth surface for efficient, long-lived devices. The benefits of adding smoothing layers in barrier structures will be discussed in Section V.

There are a number of other considerations regarding substrate selection for OLEDs on polymer substrates, including

Young's modulus, thermal stability, UV stability, dimensional stability, compatibility with process chemicals, moisture uptake, and cost. The relative importance of these is strongly dependent on the application and display structure. For a polymer/inorganic multilayer structure, the permeability of the substrate is not as significant in the effectiveness of the barrier system as the polymer used between the inorganic layers.

C. Device Layers

The anode in an OLED is typically indium-tin-oxide (ITO), though other materials such as metals, polymers, and alternative oxides have been used [25]–[27]. In a standard OLED architecture, ITO is the only brittle device layer. The conventional method of depositing ITO onto rigid substrates such as glass is by sputtering, which is a conformal deposition process and as such replicates the surface roughness of the substrate onto which it is deposited. This highlights the importance of developing a barrier structure that provides an extremely smooth surface for the deposition of device layers.

A wide range of small molecule and polymer organic materials and deposition techniques have been used in OLEDs [28]–[36]. For deposition techniques onto plastic substrates that require the presence of a solvent, e.g., ink-jet printing, the effects of any solvent interaction with the substrate and/or the permeation barrier beneath the anode must first be assessed.

Active in materials with low glass transition temperature (T_g) may be susceptible to morphological changes over time that may reduce the lifetime of the device. Any degradation processes can therefore be accelerated when the temperature of the organic layers is raised during any subsequent fabrication steps such as cathode deposition and encapsulation. The organic layers and the cathode may also be damaged or changed by any additional interaction with any solvents or vapor applied to the device post their deposition [37], or by energetic particles or radiation during processes such as sputtering or electron-beam evaporation [38]–[40]. These conditions impose further constraints upon the encapsulation method chosen when monolithic thin film barriers are used.

The cathode is typically a low work-function metal or alloy that facilitates the injection of electrons into the organic material adjacent to it. Deposition of the cathode is usually by thermal evaporation, e-beam evaporation, or sputtering. Low work-function metals that have been used include Li [41]–[44], Mg [9], In, Ca [46], and Ba. Al is often used as an additional protective layer over the low work-function layer of choice. Low work function metals are reactive by their nature and therefore require careful encapsulation.

D. Alternative Device Architectures

Described above are examples of typical materials used in traditional bottom-emitting OLEDs, i.e., in a device architecture where light exits through a transparent anode that is in intimate contact with a transparent substrate, e.g., glass or plastic. Alternative device architectures are also possible, i.e., top-emitting OLEDs (TOLEDs). Here, the cathode is transparent, thereby allowing light to exit though the top of the device [38], [39], [49]–[53]. Transparent displays can be made if the anode is also
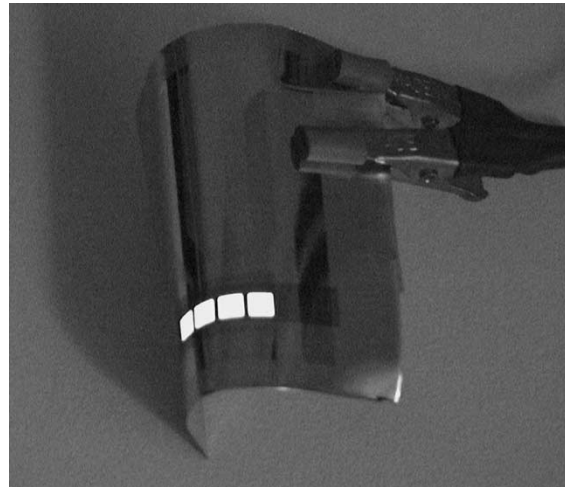

Fig. 2.   5-mm^2 TOLEDs fabricated on a stainless steel substrate.

transparent [54]. Particularly in the case of active-matrix displays, if a top-emitting architecture is employed, the fill factor can be markedly increased, which enables higher resolution displays [55] to be realized and reduces the current density requirement per pixel. This is true for both standard TOLEDs, where the anode is in contact with the substrate, or inverted OLEDs, where the cathode is in contact with the substrate. These two architectures are suited for active matrix backplanes with p-type and n-type thin film transistors respectively.

For FOLEDs, the use of top emission devices does not require that the substrate be transparent. Therefore, opaque or colored plastic substrates that have superior thermal and dimensional properties can be used. For example, polyimides such as Kapton E foil [56] have a $T_g > 350$ °C and a coefficient of thermal expansion of 12×10^{-6}/°C. Metal foil can also be employed as a substrate. Fig. 2 shows an example of a TOLED fabricated on a stainless steel substrate, which requires no barrier beneath the anode.

E. Active- and Passive-Matrix Display Architectures

Displays based on OLEDs may be addressed either passively (PMOLED) [57] or actively (AMOLED) [58], and the different architectures for each result in different encapsulation challenges. AMOLED backplanes typically contain several transistors per pixel, so the backplane processing must be compatible with the underlying barrier layers.

For PMOLEDs, the ITO lines are usually patterned by lithography. The metal cathode lines are then patterned using either a shadow mask for low-resolution displays or by incorporating an integrated shadow mask (ISM) [59] onto the surface of the substrate for higher resolution displays. The structure of the ISM can pose additional challenges to the encapsulation process. The overhang of the ISM provides a break in continuity in the organic and metal layers, creating the cathode lines. However, continuity is required for the top barrier layer. If monolithic thin film barriers are used, the barrier structure must either be sufficiently conformal, e.g., deposited via a chemical vapor deposition process, or a sufficiently thick smoothing layer must be used before fabrication of the barrier structure.

III. OLED Permeation Requirements

Permeation barriers for OLEDs are required because the devices degrade in the presence of atmospheric gases, primarily moisture and oxygen. The reactive low work function metals used as cathodes are unstable in the presence of these species and can delaminate from the underlying organic layer, and commonly used organic materials can form nonemissive quenching species upon exposure to water [60], [61]. Encapsulation of the devices and isolation of the organic/cathode materials from atmosphere has proved to be an effective technique for prolonging the lifetime of OLEDs [10].

The most widely quoted value for required water vapor transmission rate (WVTR) for an OLED lifetime of $> 10\,000$ h is 1×10^{-6} g/m^2/day. This value was originally estimated by calculating the amount of oxygen and water needed to degrade the reactive cathode [17]. Subsequent experiments comparing device lifetime to measured permeation rate have confirmed this to be a reasonable estimate [62]. Required oxygen transmission rate (OTR) for similar lifetimes has been reported as anywhere from 10^{-5} to 10^{-3} cm^3/m^2/day [18], [63], [64]. As compared to LCDs, which require a substrate barrier with an OTR of 0.1 cm^3/m^2/day and water WVTR of 0.1 g/m^2/day [47], [65], the requirements for OLED permeation barriers exceed what is available in existing substrate materials.

IV. Measurement of Permeation Rates

There are several commonly used ASTM standards for measurement of gas transmission rate, permeance, and permeability of gases and water vapor, including D1434 and D3985 for gases, and E96 and F1249 for water vapor [19]. However, none of the commercially available systems based on these techniques meet the sensitivity requirements for the low permeation rates required for OLEDs. The minimum measurable transmission rates for oxygen and water in commercial systems are 5×10^{-3} cm^3/m^2/day and 5×10^{-3} g/m^2/day, respectively. At the time of this publication, test systems exist that are capable of measuring one order of magnitude lower than this, but are available only as a service and not as a commercial instrument [66]. Development of sufficient barriers for OLEDs requires the ability to reliably measure WVTR at least as low as 10^{-6} g/m^2/day. Therefore, new techniques have been developed. One approach is known as the "Ca test" or "Ca button test." This involves observation of the optical changes as an opaque reactive metal, such as Ca, converts to a transparent oxide or hydroxide salt [63], [64], [67]. Reported effective transmission rates for H_2O through barrier films using the Ca test are as low as 3×10^{-7} g/m^2/day [63]. 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 implies that the sensitivity for WVTR reported in [63] is an upper limit since other permeants could have been present. Permeation techniques using an ultrahigh vacuum chamber outfitted with a residual gas analyzer have been used along with a calibrated aperture to obtain OTR measurements as low as 1×10^{-6} cm^3/m^2/day [68]. Performing the same measurements for water are much

more difficult due to the longer pumping times required for water. Other studies have used the percentage of inactive OLED pixel area to calculate WVTR [62], but this technique requires significant development work and does not provide a direct measurement of permeation rate. Clearly, the development and understanding of multilayer permeation barriers will be aided by improved measurement techniques.

V. Thin-Film Barrier Technology

Before the development of OLED technology, the majority of thin-film barrier research was for the food and pharmaceutical packaging industries. Based on that research, some of the general concepts in permeation barriers on polymers will be addressed, followed by results specific to OLED technology.

A. Permeation Through Barrier Coated Polymers

Food and packaging applications can require $100 \times$ improvement in barrier properties over what is provided by uncoated polymers [23]. Inorganic coatings have been used since the 1960s to reduce the permeation rates through polymers. Organic-inorganic hybrid polymer coatings (ORMOCERs and ORMOSILs) have also been used [69]–[71]. Evaporated aluminum films on PET were commercialized in the early 1970s. Dielectric films, typically SiO_x or SiN_x, offer microwaveability and transparency, and are now commonly used for food packaging. The best single-layer barrier performance is achieved for dielectric films deposited by plasma-enhanced chemical vapor deposition (PECVD) [20], [72]. An excellent review of the performance of various films for food packaging is given by Chatham [23].

Bulk oxides and aluminum are effectively impermeable to O_2 and H_2O [73], as are perfect SiO_2 films [74], [75]. However, single barrier layers provide at best only two to three orders of magnitude improvement over the O_2 transmission rates of the polymer substrates [23], [48], [72], [74], [76]–[78]. This is caused by permeation through defects or pores rather than the barrier film, and explains the surprising observation that oxygen permeation rate for single layer barriers on polymer substrates generally changes by no more than an order of magnitude irrespective of the coating method or material [23]. Even though the surface area fraction of defects is small, lateral diffusion causes the total permeation rate through many small pinholes to be much higher than if the pinhole area were combined into fewer, larger defects [79], [80]. Lateral diffusion results in a large concentration gradient of permeants near the pinhole, such that increasing the barrier performance of the polymer immediately adjacent to a pinhole defect significantly improves the overall barrier performance [48], [81].

Evidence for the defect-driven permeation mechanism is found in the analysis of activation energy for diffusion. Several studies have shown that for single-layer barriers, even for high quality films, the activation energy for oxygen permeation is characteristic of diffusion through the substrate, regardless of the coating material or deposition technique [74], [77], [78], [82]. It is also seen in the correlation of film-defect density with permeation rates. Fig. 3 shows combined data from da Silva Sobrinho *et al.* and Jamieson and Windle for the OTR
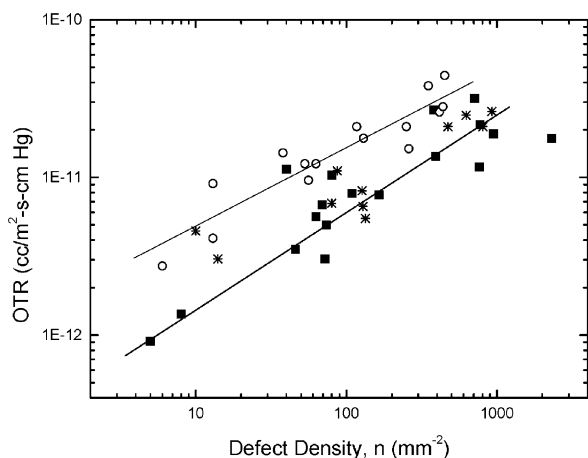
Fig. 3. OTR as a function of defect density for $Al(\circ)$, SiO_x $(*)$, and SiN_x $(\blacksquare)$ films with thickness $> \Lambda_c$. Note that 1.5×10^{-12} $cc/m^2/s/cm$ Hg is the equivalent of 0.1 $cm^3/m^2/day$. (Reproduced with permission from [74]).

Fig. 4. Defect density $n(\triangle)$ and OTR $(\blacksquare)$ versus coating thickness for PECVD-deposited SiO_2 on 13-μm PET. Note that 1.5×10^{-12} $cc/m^2/s/cm$ Hg is the equivalent of 0.1 $cm^3/m^2/day$. (Reproduced with permission from [74]).

of SiO_x, SiN_x, and Al-coated PET versus defect density, clearly demonstrating the dependence between the two. Defect densities for Al films was measured using optical microscopy, while for transparent films, a novel approach using reactive ion etching was used to define the defects [48], [72], [76]. Complex simulations of two-dimensional diffusion [79], [81], finite-element analysis [48], [80], and simple geometric models [74] have been used to successfully correlate pinhole density with permeation rate.

There is some disagreement over the source of defect-driven permeation through plasma-deposited SiO_x films. The correlation between defect density and the oxygen transmission rate shown in Fig. 3 was attributed to pinhole defects with a size distribution centered around 0.6 μm [74], comparable to the 0.7 and 1–2 μm pinholes found in Al films on BOPP and PET substrates, respectively, in different studies [23], [80]. Both were attributed to particulate contamination or surface roughness of the substrate. In other studies, permeation through SiO_x films has been attributed to morphology effects. Erlat has shown that lacking the presence of pinhole defects permeation rates still reduce by less than three orders of magnitude over the substrate alone [77]. For SiO_x, Al, and AlO_x films, the O_2 permeation rate was shown to be related to surface roughness of the barrier film, indicating that pinhole defects are not the only source of permeation through barrier films [77], [82], [83]. Henry et al. observed no evidence for pinholes; the activation energy for permeation was not characteristic of the substrate and no dependence on morphology was observed. A pore-size distribution of 2.7–4 Å was used to model permeation [84]. Transmission electron microscopy showed that poor barrier films can have intergranular defects, typically 4–6 nm in diameter, covering as much as 30% of the sample area, while good barrier films exhibit no defects implying that if the defects are present they are at least $<$1 nm in diameter [77]. Dennler et al. have used scanning electron microscopy to show a high density-defect distribution of pinholes with diameters of tens of nanometers for SiO_2 films with thickness in the 1–10-nm range (below Λ_c, which is discussed in the next paragraph) [85].
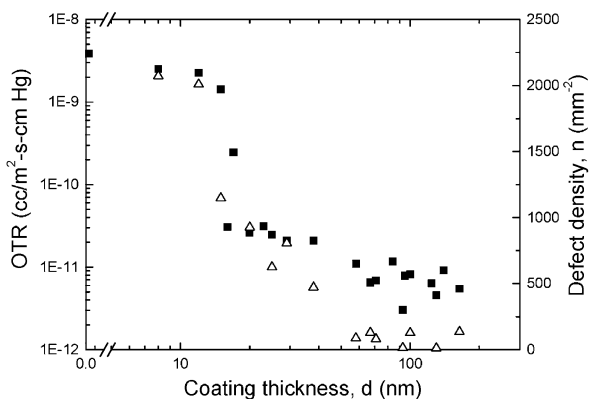
A simple method for understanding the permeation behavior of a film/substrate couple is to study the barrier performance as a function of film thickness [23]. Thermally activated diffusion, or "solubility-diffusion" permeation, through a bulk material exhibits a characteristic thermal activation energy, and permeation varies inversely with the thickness of the film [23], [73], [86]–[88]. However, this is not normally seen for thin-film barrier layers. A common behavior for gas transmission as a function of film thickness is a gradual reduction for very small thicknesses, followed by a rapid decrease of about two orders of magnitude, followed by a slow decrease to a minimum value, ρ_{min}, as the film thickness increases further. The thickness at which the rapid decrease occurs is called the critical thickness, Λ_c. This characteristic behavior is evident in Fig. 4 from da Silva Sobrinho et al., which shows OTR versus coating thickness for SiO_2 films [74]. Thus, significant improvement in the transmission rate can only be achieved if the inorganic layers exceed Λ_c. The value of Λ_c is dependent on both material and deposition techniques. For evaporated Al [23] and plasma-deposited SiO_2 and SiN_x [74], Λ_c is $<$15,, 12–15, and 8 nm, respectively [23], [74]. A review of the literature showed that, surprisingly, there is only a small $(<10x)$ dependence of ρ_{min} on material or deposition technique [23]. There was found to be a large dependence of ρ_{min} on substrate material, consistent with the defect-driven permeation model discussed previously. In contrast to speculation that films with thickness $< \Lambda_c$ were discontinuous, it has been shown that films as thin as 2 nm are continuous but contain a large defect density, consistent with the intergranular nanopore defect model [72], [85].

Other inorganic materials have been investigated for barriers. The higher density of silicon nitride results in better barrier performance for this material than for SiO_x films, including reduced Λ_c and reduced sensitivity to deposition parameters [4], [72], [89]. Sputtered silicon oxynitride was shown to be an effective barrier, combining the good barrier properties of SiN_x with the transparency of SiO_x films [4]. Numerous materials have been evaluated using e-beam evaporation as the deposition technique, including the oxides of Ce, Na, Si, Mg, Al, Zr, Ti, and Ta, as well as MgF_2, but the barrier performance was too poor for OLEDs [47], [90], [91]. Sputtered dielectric barrier

films have included Al, Ta, and Si oxides [47], [82]. Plasma-deposited diamond-like carbon films have been evaluated for barrier performance, but were not as effective as the SiO_xN_y class of materials [92]. Vapor- and plasma-polymerized polyxylenes have been used to encapsulate OLEDs, but the performance improvement was also minimal [93], [94]. Sputter-deposited hydrocarbon films have shown a factor of >100 improvement in the barrier performance of 12-μm PET films, while still able to undergo 2.8% strain before the onset of microcracking. However, optical transmission decreased from 83% to 73% for the best barrier performance [95].

It should be noted that many barrier studies measure only OTR or WVTR, but barrier performance is largely dependent on the permeant species. Permeation in polymers occurs by different mechanisms for nonpolar, noninteracting molecules such as oxygen than for polar, condensable molecules such as water that may interact with the barrier film and polymer [77].

B. Polymer-Inorganic Interfaces in Barrier Structures

Numerous techniques have been used to study the interfaces between polymer substrates and deposited inorganic layers. It was shown that even for evaporated SiO_x films on PET, the interface is composed of strong covalent bonds, comprised of Si-C and Si-O-C bonds. For less reactive polymers, such as polyethylene, N_2 plasma treatment of the substrate is required prior to deposition to get uniform coverage, and leads to Si-O-C and Si-N-C bonding at the interface [96]. The interface between PET and PECVD SiO_x and SiN_x films is quite different than for PVD films [97], [98]. For the plasma deposited films, there exists an "interphase" region of 40–100 nm for both SiO_x and SiN_x. This region has a crosslinked organosilicon nature and exhibits a continuously graded composition between the PET substrate and the inorganic layer. The interphase region provides improved mechanical stability and stress relaxation across the interface. This interphase region is no more than several nanometers for PVD films.

C. Multilayer Barrier Structures

The best performance from single-layer barrier structures is orders of magnitude short of the requirements for OLEDs, but significant improvement in permeation-barrier performance can be achieved by using multilayered structures. Successive deposition of different inorganic films improves barrier performance only slightly [77], [78], [99]. However, interesting synergistic effects occur with polymer/inorganic multilayer structures. This is evident from the data in Table II [100]. While a polyacrylate film only improves the oxygen-barrier performance of a polymer substrate by a few percent [101], a polyacrylate layer deposited prior to aluminum metallization provides a 15x improvement in O_2-barrier performance over aluminum without the underlying polyacrylate layer. This has been attributed to smoothing of the substrate, reduced mechanical damage, increased thermal stability of the nucleation surface, and increased chemical polarization of polyacrylate over polypropylene. Interestingly, a polyacrylate layer on top of the polyacrylate/aluminum stack yields another order of magnitude improvement in barrier performance. This was attributed to improved abrasion resistance of the top coating

TABLE II
OTR FOR POLYPROPYLENE WITH VARIOUS COATINGS [100]

BARRIER SYSTEM	OTR (cm^3/m^2/day)
Polypropylene (PP)	~1500
PP/polyacrylate (PA)	~1400
PP/aluminum (Al)	~30
PP/PA/Al	~0.8
PP/PA/Al/PA	~0.08

[100]–[102]. Leterrier has also noted that a polymer top coat can improve the mechanical ruggedness of an inorganic layer by passivating mechanical flaws in the inorganic layer [20]. This polymer multilayer (PML) process was first developed at GE for capacitors when it was observed that the number of pinholes in metal films was significantly reduced [103]. Similar barrier results were obtained using transparent dielectric layers rather than Al, including both reactively sputtered and electron-beam evaporated Al_2O_3 [101], [103]. The smoothing effect of the polyacrylate occurs due to the unique deposition process in which the flash evaporated liquid acrylate monomer first condenses on the substrate surface and is subsequently cured by UV rays or an electron beam [100], [101]. Similar planarization has been achieved with spin-coated UV curable resins [3], [4]. These promising results led to multilayer structure with multiple polymer/inorganic 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 PML structure. By varying the total number and thickness of the polymer and inorganic layers in the thin-film coating, both the optical and barrier properties of the barrier can be tailored. Barrier structures were demonstrated achieving WVTR estimated to be as low as 2×10^{-6} g/m^2/day using the Ca test [64]. Results for OLED devices with these multilayers will be presented in Section V-D.

D. Barriers for OLED Devices on Flexible Substrates

Applying thin-film barrier encapsulation to OLED displays is especially challenging. The barrier requirements for OLED displays fabricated on rigid substrates, e.g., glass, should possess the following characteristics, at a minimum.

- The deposition process must be compatible with OLEDs, i.e., must not damage the active components in the display.
- The permeation rate of water through the barrier must meet the requirements discussed earlier, i.e., WVTR $< 10^{-6}$ g/m^2/day [17] and OTR $< 10^{-3}$ cm^3/m^2/day.
- The barrier, or layer(s) covering it, must be mechanically robust, i.e., tough enough to allow the user to handle the display without loss of barrier performance.
- The barrier must be stable for the lifetime of the display, i.e., exhibit good adhesion to the display surface and a similar thermal expansion coefficient to the layers beneath.
- The barrier must be resistant to any processes, e.g., lithography, that are carried out on it during the fabrication of the OLED display.

In the case of FOLEDs, in addition to the above requirements additional properties are required that include.

- The barrier must be flexible.

Fig. 5. 128×64 passive matrix (60 dpi) monochrome green PHOLED display fabricated on barrier coated 0.175-mm heat stabilized PET (courtesy of Universal Display Corp.).
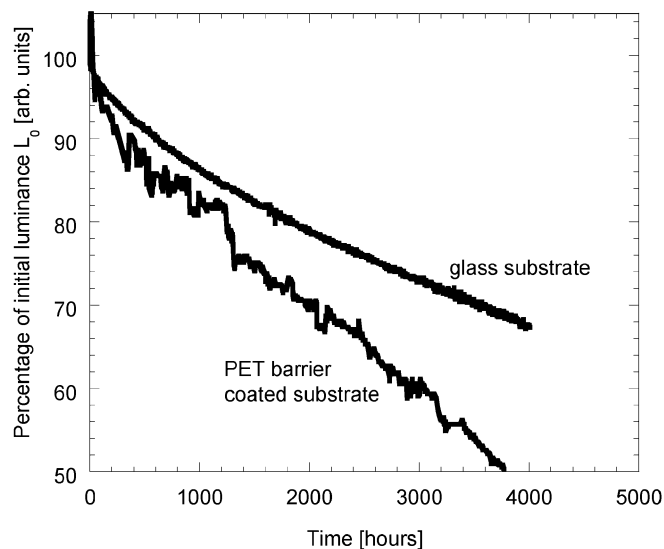

Fig. 6. Room temperature lifetime plots of two PHOLEDs, the first deposited on barrier coated PET driven at 2.5 mA/cm^2 and for comparison a device with the same architecture fabricated on a glass substrate driven at 2.6 mA/cm^2 [62].

- The barrier must retain its barrier properties as a function of in-flex use during the lifetime of the display.

For FOLED displays to satisfy the above requirements, the most common approach used to date has been to use a PML structure on both the substrate and over the OLED display. Harvey *et al.* [15] at Motorola and Affinito *et al.* [16] and Graff *et al.* [104] at the Pacific Northwest National Laboratory, have all proposed using multilayers composed of alternating oxide and polyacrylate layers. Vitex Systems Inc. is working to exploit this approach both as a barrier coating for flexible plastic substrates and laminated lids, and as a final encapsulation barrier over the OLED display surface [105]. This approach takes advantage of the improved barrier quality of organic/inorganic multilayers mentioned earlier.

1) OLED on Barrier-Coated Substrate: The first demonstration of a FOLED display built on a flexible PML substrate (see Fig. 5) was by Burrows *et al.* [17]. A 128×64 passive matrix (60 dpi) monochrome display was fabricated on barrier coated, 0.175-mm-thick heat-stabilized PET. Weaver [62] fabricated 5-mm^2 high-efficiency phosphorescent OLED (PHOLED) test pixels on a similar barrier-coated substrate. The PHOLEDs had comparable electrical characteristics to equivalent devices made on glass substrates. A WVTR through the plastic substrate of 2×10^{-6} g/m^2/day was estimated by encapsulating the devices using a conventional glass lid and incorporating calcium-oxide desiccant. This value is in close agreement with the permeation rate measured using the calcium button test [63]. The OLEDs were driven at 2.5 mA/cm^2 (425 cd/m^2) dc and a device lifetime of 3800 h (defined as the time taken to decay to 50% of its initial luminance) was measured (see Fig. 6). While 3800 h is not an adequate lifetime for commercial displays, this work demonstrated an order of magnitude increase in lifetime over other substrate barrier approaches and showed the feasibility of this approach as a substrate barrier for FOLEDs.

2) OLED with Monolithic Thin Film Barrier: Akedo *et al.* have demonstrated monolithic thin film encapsulation on a glass substrate for a 9-mm^2 test device. The encapsulation was a PECVD SiN$_x$/ plasma-polymerized CN$_x$:H multilayer stack,
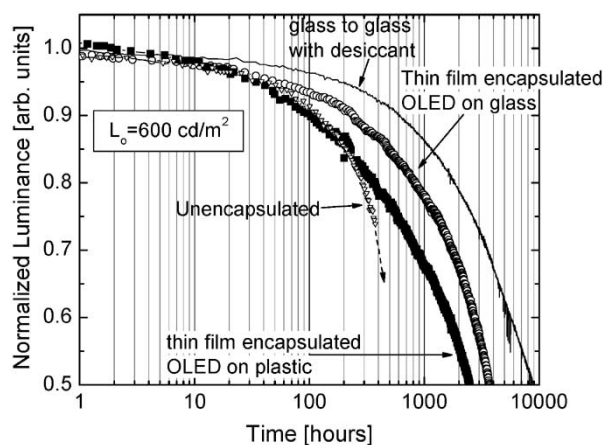

Fig. 7. Normalized room temperature lifetime data for 5-mm^2 PHOLEDs under dc constant current drive from a starting luminance of 600 cd/m^2. Plotted are an unencapsulated PHOLED on glass ($\triangle$), a thin-film encapsulated PHOLED on PET ($\blacksquare$), a thin film encapsulated PHOLED on glass ($\bigcirc$), and a PHOLED on glass packaged with a glass lid and desiccant (solid line). Half lives of the devices are <600, 2500, 3700, and 9100 h, respectively [6], [7].

and the test device demonstrated a lifetime of >1000 h at 85°C [106].

Chwang *et al.* [6], [7] have included a multilayer barrier over the OLED surface, in addition to beneath it on the plastic substrate. Again, a similarly barrier coated 175-μm-thick PET was used as the substrate. PHOLED test pixels 5 mm^2 in area were deposited onto the substrate. The FOLED was then encapsulated using a PML structure consisting of four or five pairs of alternating polyacrylate and Al$_2$O$_3$ layers. The total thickness of the multilayer-thin film coating was less than 7 μm. Fig. 7 shows the lifetime of these devices tested from a starting luminance of 600 cd/m^2. For comparison, lifetime data are shown for devices with no encapsulation, thin-film top barrier with a glass substrate, and conventional encapsulation with dessicant on glass substrate. The PHOLED with the standard glass package shows

a lifetime of >9000 h. The PHOLED with the thin-film encapsulant and glass substrate decays to 50% luminance in 3700 h, i.e., ~41 % of the time taken by the traditionally encapsulated PHOLED. The lifetime observed from the PHOLED test pixels on the barrier coated plastic with thin-film encapsulation was 2500 h.

As has been discussed previously, the complexity of fabricating a FOLED display is greater than for test pixels. The number of lithography steps is greater, there is a more complex topography on the display backplane and the increased surface area over which pinhole free high barrier encapsulation must be achieved is a significant challenge for the encapsulation process.

In 2003, Chwang *et al.* reported the first lifetime results from a thin-film barrier-coated display [6]. The group reported fabrication of a monochrome, 64×64, 80 dpi passive-matrix FOLED encapsulated in the same way as the 2500-h 5-mm^2 test devices discussed previously. From an initial luminance of 110 cd/m^2 (a passive-matrix duty cycle of 1/64), the drive-display lifetime was measured to be 200 h. Similar displays that were not driven exhibited aging, and had a shelf life of >700 h. One major cause cited for the reduced lifetime over the test coupons was the presence of a photoresist ISM. The bake conditions of the grid were not optimal due to the thermal limitations of the PET substrate, and residual solvent was thought to be a potential reason for the shorter lifetime. Nevertheless, this lifetime demonstrated a first step in producing long-lived OLEDs on plastic.

Recently, Yoshida *et al.* [3], [4] used a similar multi-layer-barrier approach by spin coating a UV curable resin onto a plastic substrate to planarize the surface. They then deposited a SiON moisture-barrier film and repeated the resin/SiON layer pairs to achieve the desired barrier performance. A full-color 160×120 $3''$-diagonal FOLED display was then fabricated on the barrier-coated substrate, avoiding the complications of the ISM by using shadow masks to define the organic and cathode layers. The display was subsequently encapsulated with a PECVD SiN$_x$ film. Test pixels grown, using the same architecture, demonstrated a projected half lifetime of 5000 h from an initial luminance of 1000 cd/m^2, but no lifetime was reported for the display.

E. Getters for Moisture Absorption in Flexible Devices

As shown in Fig. 1(a), moisture gettering material is normally included during device encapsulation to extend the lifetime of the device. This getter material is typically at least a fraction of a mm thick. From a mechanical perspective incorporation of a millimeter-thick getter is not possible for flexible devices. However, it is possible to consider incorporation of a moisture gettering thin-film layer. Examples of how the thin-film getter layer might be incorporated into a monolithic barrier structure or a laminated barrier structure are shown in Fig. 8(a) and (b), respectively. The extended lifetime of an OLED due to getter incorporation is a function of the getter coverage and the volume of water that can be absorbed. While it is likely that coverage can be improved in a thin-film device by coating the entire device area with a getter, the volume of getter is significantly reduced.

The thickest layer that can be mechanically tolerated would be the most effective.

The effect of the getter layer on the permeation-barrier layer must be considered. Getter materials swell when water is absorbed. For a monolithic barrier structure, such as the one in Fig. 8(a), local swelling could cause local failure in the barrier layers and premature degradation. Barrier-layer failure due to swelling would not be a major concern for a laminated device. Using a structure similar to the one in Fig. 8(b), Tsuruoka *et al.* demonstrated the incorporation of a thin-film getter into a device on a glass substrate. Significant reduction in dark-spot formation and pixel shrinkage was shown [107]. However, this particular getter required higher curing temperatures than are possible for inexpensive polymer substrates. Incorporation of thin-film getters into flexible devices is an intriguing idea that, to our knowledge, has yet to be demonstrated.

A third concept for incorporating a getter material into a flexible device has been described by Duggal [108]. This method involves embedding gettering particles into the polymer substrate and/or a polymer lid. The gettering particles need to be sufficiently less than the wavelength of emitted light (preferably $<1/5$) to not scatter the light. Barrier layers are then fabricated on the external surfaces of the substrate, as shown in Fig. 8(c). This method allows a larger volume of gettering particles than just a thin film, as the entire substrate volume is available for filling.

VI. MECHANICAL FLEXING OF THIN-FILM PERMEATION BARRIERS

It has been established that effective barrier systems for FOLEDs will include inorganic thin films. For a display to be flexible, the display, in addition to the requisite barrier layers, must be able to withstand the repetitive stress and strain of being flexed and bent by the user. For transparent permeation barriers, the inorganic layers will likely be a dielectric oxide, nitride, or some similar brittle material. For these films, cracking due to mechanical flexing can render the barrier film ineffective. The mechanical effects of flexing are determined by the particular display architecture, but in all cases, one must consider the tensile and compressive stresses in the barrier layers, shear stress in adhesive layers, abrasion between laminated layers, and adhesion strength between thin film layers.

The mechanics of thin films on rigid substrates are well understood [109], but for rigid films on flexible substrates the mechanics change considerably. A significant amount of research has been devoted to understanding the electrical and mechanical response of ITO thin film under stress because this is the only inorganic active layer in a typical device [110]–[113]. In fact, experiments on OLEDs under bending have shown that failure occurs in the ITO layer [114], which cracks under a tensile strain of $<2.5\%$ [112]. Cyclic loading of ITO has shown that after repeated flexing, ITO cracking can occur for a lower tensile strain of just 1.5% [110]. However, it is important to note that the mechanical requirements for barrier layers are much more demanding than for device layers such as ITO. While microcracks in ITO lead to undesirable increased resistance, microcracks in barrier layers result in barrier failure.
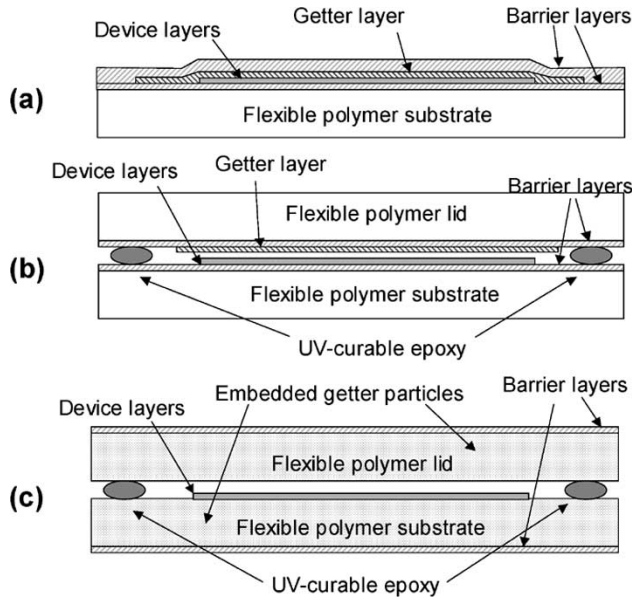
Fig. 8. Potential structures for incorporation of: (a) a thin-film getter into a monolithic barrier structure; (b) a thin film getter into a laminated barrier structure; or (c) a particulate getter into a polymer substrate.

Cracking of silica-barrier films has been studied and the mechanical properties of SiO_x films have been measured [115]–[117]. Yanaka *et al.* have studied evaporated SiO_x films on PET as a function of temperature. They found that while crack density decreases with higher temperature, the onset strain for crack formation was 1.3%, regardless of the temperature [118]. Leterrier measured a value of 1.2%–2.0%, depending on the coating thickness [119], which is similar to the value observed for ITO. A useful analysis of the mechanics of thin films on flexible substrates has been given by Suo *et al.* [120]. For uniform deformation (film has the same Young's modulus, Y_f, as the substrate, Y_s), the strain at the top surface is given by

$$\varepsilon_{\text{top}} = \frac{(d_f + d_s)}{2R} \tag{1}$$

where d_f and d_s are the film and substrate thicknesses, respectively, and R is the radius of curvature.

When a brittle film is used on a pliable substrate, the neutral plane, where no compressive or tensile strain exists, shifts toward the film, reducing stress in the film. The resulting film strain is

$$\varepsilon_{\text{top}} = \left(\frac{d_f + d_s}{2R} \right) \frac{(1 + 2\eta + \chi\eta^2)}{(1 + \eta)(1 + \chi\eta)} \tag{2}$$

where $\eta = d_f/d_s$ and $\chi = Y_f/Y_s$. For example, using values of 5.53 and 73 GPa for the moduli of PET [111] and SiO_x films [118], respectively, and 100 μm and 100 nm for the substrate and film thickness, respectively, a strain of 1.3% is reached at a radius curvature of 4 mm. This is a simplified example, as multilayer composite films make the analysis more complicated, and analysis will have to be specific to the particular barrier/substrate system. The governing equations for elastic stresses in multilayered thin films on a thick substrate have been

developed by Townsend *et al.* [121], and a thorough discussion of mixed-mode cracking in layered materials was given by Hutchinson and Townsend [122].

Improvements in the mechanical response of a barrier/substrate system can be achieved by placing the barrier layers at or near the neutral plane, such that strain is minimized. One approach is to use a laminated structure which is identical to the device substrate, as shown in Fig. 1(a) [18]. In this case, failure of the device will more likely be determined by the substrate itself, or by shear stress in the adhesive. In this type of structure, polymer substrates offer an advantage over thin glass by providing a lower modulus and a higher degree of flexibility before substrate failure. One approach for improving the mechanical robustness of monolithic barrier systems is to use a high-modulus layer above the barrier layers with properties tailored for the substrate/barrier system such that the neutral plane is at or near the barrier layers. Another interesting alternative is to insert a lower modulus buffer layer between the substrate and the barrier layers, which helps to move the neutral plane toward the barrier layers, as shown by Park *et al.* [111].

Several authors discuss the importance of residual stresses in a thin film in minimizing cracking [20], [123]. Internal stresses can lead to nanoscale cracking for thicker films, forcing a compromise between barrier performance and mechanical robustness. Compressive film stress, in addition to improving tensile strength and adhesion, provides better barrier performance for PECVD-oxide layers [20], [124]. Leterrier concludes that compressive internal stresses improve the film density, barrier performance, coating tensile strength, and coating/substrate adhesion for a barrier system. Regarding the stability of the film stress and the coating strength, water uptake in a SiO_x-barrier film will reduce the compressive internal stress, and a SiN_x-capping layer can significantly improve the internal stress stability [20]. However, water uptake in the polymer substrates will also cause swelling and affect the stress state of the barrier film. In addition to film stress, coating strength is strongly affected by surface flaws. Therefore, surface roughness is critical. Polymer sizing of surface flaws may partially explain why polymer/oxide/polymer multilayers show improved performance.

Several studies have shown that both polymer and small-molecule OLEDs operate with little or no reduction in performance during bending down to a certain radius. For OLEDs on 125 μm PET a bending radius of 15 mm does not affect device performance but no barrier layers were used, and no degradation performance was reported [114]. Chwang *et al.* have demonstrated the effects of flexing on a passive-matrix FOLED, but effects on barrier layers and degradation were not reported [6], [7].

VII. OPTICAL CONSIDERATIONS

An effective barrier system will consist of at least one substantially transparent layer between the device and the viewer, and will likely contain a multilayer stack. Therefore, it is important to consider desirable or undesirable optical effects created by the barrier system. Transparency is a major consideration, as this directly influences the efficiency of the display. A compromise, therefore, needs to be found between the transparency of

the barrier film across the visible spectrum and its permeability to water and oxygen. For example, Yoshida *et al.* [4], [125], used silicon oxynitride (SiON) as a permeation barrier and varied the oxygen-density ratio $(O/O + N)$ in order to optimize the films optical properties (transparency $>90\%$) with adequate moisture impermeability for OLEDs.

Optical microcavity effects are caused by interference between at least two partially reflecting surfaces separated by distances on the order of the wavelength of light being studied. For wavelengths experiencing constructive interference, this can lead to spectral narrowing and intensity enhancement. This allows a properly designed cavity to have improved color purity, but at the expense of spectral shifts and intensity variation as a function of viewing angle. For microdisplay applications where the viewing angle is fixed to a narrow range, microcavity effects can be a useful tool. However, for most display applications, color shifts as a function of viewing angle are undesirable. Therefore, it is important to model the entire device structure, including the barrier system, to ensure that microcavity effects are understood and controlled. Several studies specific to OLEDs have modeled and experimentally demonstrated microcavity effects [126]–[135]. Most of these studies used a distributed Bragg reflector, or dielectric mirror, external to the device layers. Alternating barrier layers can have similar optical effects, and these must be considered when designing a multilayer permeation barrier. One report has modeled Al_2O_3/polymer multilayers and showed that the interference effects for the particular system used were small [136].

VIII. CONCLUSION

The realization of FOLED displays requires further advances in the technology of thin-film permeation barriers. Several barrier architectures are possible and each has different processing, mechanical, and optical considerations. Improved permeation-rate measurement systems for ultrahigh barrier films are needed. Single barrier-layer structures are not sufficient for OLEDs, but much of the thin-film barrier science can be learned from these structures and applied to multilayer structures. The synergistic effects of polymer and inorganic multilayer structures appear to be the most promising approach for encapsulating flexible displays whether used as a monolithic thin film coating over the OLED, or on a laminated flexible lid. Better understanding of the synergistic effects of polymer/inorganic multilayers will lead to improved barrier performance from simpler structures and it is clear that more studies on the effects of mechanical stress on barrier performance and device lifetimes are needed. Recent results show device lifetimes of 3800 h from an initial luminance of 425 cd/m^2 using a thin-film barrier on a PET substrate [62], and another study demonstrated a projected half life of 5000 h from an initial luminance of 1000 cd/m^2 for a device on a flexible substrate [4]. This early work shows significant promise in helping realize a truly flexible display medium.

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Jay S. Lewis received the B.M.E. degree from the Georgia Institute of Technology, Atlanta, in 1996 and the M.S. and Ph.D. degrees in materials science and engineering from the University of Florida, Gainesville, in 1998 and 2000, respectively.

He is currently with the Microelectronics Center of North Carolina (MCNC) Research and Development Institute, Research Triangle Park, as a Research Scientist. He was previously with Hewlett Packard Labs researching cathodoluminescent phosphors, and Planar Systems, Inc., researching novel phosphors for inorganic electroluminescence. His current areas of research involve optoelectronic devices and materials, including organic LEDs, organic electronics, and photonic crystals.

Dr. Lewis is a Member of the American Vacuum Society, the Materials Research Society, the Society for Information Display, and the American Association for the Advancement of Science.

Michael S. Weaver received the Ph.D. degree in physics from Sheffield University, Sheffield, U.K., in 1996

From 1996 to 2000, he was with the European Research and Development Laboratory, Sharp, Oxford, U.K. The two main research topics were the study of new materials and building passive matrix small molecule OLED displays. He is currently a Senior Scientist with the Universal Display Corporation (UDC), Ewing, NJ. At UDC, his research interests include PHOLEDs, top-emitting OLEDs, and FOLED displays.