Direct Encapsulation of Organic Light-Emitting Devices (OLEDs) Using Photo-Curable *co*-Polyacrylate/Silica Nanocomposite Resin

Yu-Young Wang, Tsung-Eong Hsieh, I-Ching Chen, and Chin-Hsin Chen

***Abstract*—Direct encapsulation of organic light-emitting devices (OLEDs) was realized by using highly transparent, photo-curable *co*-polyacrylate/silica nanocomposite resin. Feasibility of such a resin for OLED encapsulation was evaluated by physical/electrical property analysis of resins and driving voltage/luminance/lifetime measurement of OLEDs. Electrical property analysis revealed a higher electrical insulation of photocured nanocomposite resin film at 3.20×10^{12} Ω in comparison with that of oligomer film at 1.18×10^{12} Ω at 6.15 V to drive the bare OLED. This resulted a lower leakage current and the device driving voltage was efficiently reduced so that the nanocomposite-encapsulated OLED could be driven at a lower driving voltage of 6.09 V rather than 6.77 V for the oligomer-encapsulated OLED at the current density of 20 mA/cm^2. Luminance measurement revealed a less than 1.0% luminance difference of OLEDs encapsulated by various types of resins, which indicates that the photo-polymerization takes very little effect on the light-emitting property of OLEDs. Lifetime measurement of OLEDs found that t_{80}, the time span for the normalized luminance of device drops to 80%, for nanocomposite-encapsulated OLED is 350.17 h in contrast to 16.83 h for bare OLED and 178.17 h for the oligomer-encapsulated OLED. This demonstrates that nanocomposite resin with optimum properties is feasible to OLED packaging and a compact device structure could be achieved via the method of direct encapsulation.**

***Index Terms*—Direct encapsulation, organic light-emitting devices (OLEDs), photo-curable nanocomposite resin.**

I. Introduction

ADVANCE of flat panel displays (FPDs) has brought out many new display technologies such as organic lighting-emitting devices (OLEDs) and polymer lighting-emitting devices (PLEDs) because of their unique properties [1]–[4]. Although OLEDs and PLEDs exhibit great potential for applications, their feasibility to electronic products is restricted by several difficulties including instability of organic light-emitting materials [5], short lifetime resulting from device structures [6], immature packaging process, and sealing materials [7], [8]. Among these, hermetic sealing is crucial to OLEDs because organic light-emitted materials cannot tolerate the moisture/oxygen attack [9], [10]. In conventional bottom-emitting OLEDs, a metal lid is attached on the glass substrate containing sequential layers of cathode electrode, light-emitting materials, and anode electrodes. The sealing is accomplished by applying low-moisture permeability adhesive resin between the lid and substrate. However, such a packaging method becomes inappropriate for new types of OLEDs, e.g., flexible OLEDs (FOLEDs). Furthermore, since thinner and lighter FPDs are always desired, direct encapsulation of OLEDs, hence, becomes one of the popular research topics [8], [11]–[13].

As a result of improved physical properties such as higher thermal stability, better mechanical strength, lower permeability, and thermal expansion properties [14]–[20], nanocomposite resins have become the promising materials for OLED packaging. In addition to the feasibility of ultraviolet (UV)-curable nanocomposite resin applied to OLED packaging, in this paper we also demonstrate that a thinner, lighter packaging structure could be achieved via direct encapsulation of device utilizing the new class nanocomposite sealing resin [20].

II. Experimental

A. Preparation and Characterization of Nanocomposite Resins

The photo-curable *co*-polyacrylate/silica nanocomposite resin was prepared via *in-situ* sol-gel process, and its synthesis process and property characterization could be found elsewhere [20]. The components for preparation of this nanocomposite include the oligomer (bisphenol A epoxy diacrylate) purchased from Sartomer, the radical photopolymerization initiator (1-hydroxycyclohexylphenyl ketone) obtained from Cambridge International, and acrylic acid and tetraethyl orthosilica (TEOS) purchased from Aldrich Chemicals. The preparation and characterization of nanocomposite resin were briefly described as follows. First, TEOS and appropriate amount of acrylic acid were added in sequence into a three-necked flask containing oligomer heated at 80 °C. The whole mixture was stirred constantly for 24 h to precede the *in-situ* sol-gel reaction and the H_2O necessary for hydrolysis reaction was acquired via moisture in the ambient at about 60%RH. After the completion of sol-gel reaction, about 5.0 wt.% of photo-initiator was

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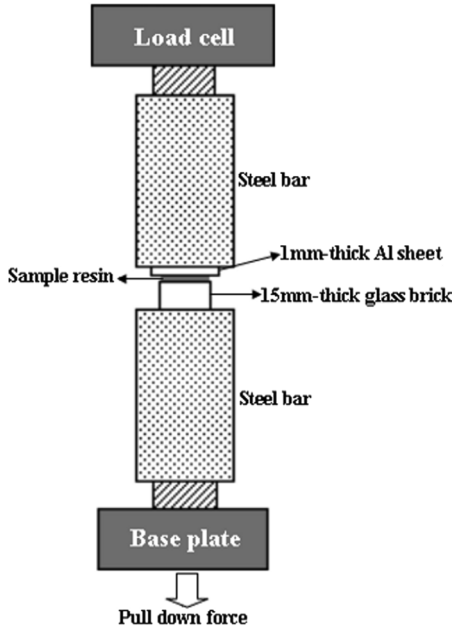

Fig. 1. Schematic illustration of adhesion test.

added into above mixture, and the stirring was further carried out for 120 min to complete the preparation of photo-curable nanocomposite resin.

Adhesion strength of resins on glass and aluminum (Al) surfaces was measured viaa pull test in accordance with ASTM D-3528 standard, and the test scheme was shown in Fig. 1. Nanoindentation tester (Hysitron Triboscope) was adopted to evaluate the mechanical properties of resin films [21], [22]. The moisture permeability was measured at 40 °C and 90%RH using a permeation detection instrument (Permatran-W 3/60, Mocon). The dynamic mechanical analysis (DMA, TA2980) was carried out at a heating rate of 5 °C/min to identify the viscoelastic properties and glass transition temperature (T_g). The dimension of DMA samples is 4 mm × 25 mm × 100 μm. A thermogravimetric analyzer (TGA, TA2950) was utilized to identify the 5.0% weight loss decomposition temperature (T_d). The chemical structures of resin samples cured at various energy densities were analyzed by a Fourier transform infrared spectrometer (FT-IR, Nicolet Protégé 460) at the wavenumber ranging from 400 to 4000 cm^{-1}. The transmittance of encapsulation resins was obtained by an HP8453 UV-visible spectrometer with scanning wavelength ranging from 190 to 1200 nm. The results of physical property characterization for oligomer and nanocomposite resins cured at 3.0 J/cm^2 (without post baking) are listed in Table I, and the thermal properties on nanocomposite resin cured at different energy density are shown in Table II. A representative microstructure of nanocomposite resin is shown in Fig. 2.

B. Electrical Properties of Photocured Resins

The samples for electrical property characterization were prepared as follows. The oligomer and nanocomposite resins were first coated on the precleaned indium–tin–oxide (ITO) glass substrate, respectively. The photo-polymerization of resins was carried in a UV oven (UVP CL-1000) in which

TABLE I
PHYSICAL PROPERTIES OF OLIGOMER AND NANOCOMPOSITE RESINS CURED AT 3.0 J/cm^2

Physical properties	Oligomer	Nanocomposite
Inorganic content (*wt.*%)	–	11.12
T_d (°C)[a]	366.5	176.7
T_g (°C)	86.2	106.8
CTE (ppm/°C)	99.3	30.6
Average transmittance in visible light range (%)[b]	82.33	81.92
Moisture permeability (g/m^2·24 hrs)[c]	13.59	10.41
Moisture absorption (%)[d]	1.148	1.013
Adhesion strength on Al (kgf/cm^2)	3.58±0.71	16.72±1.88
Adhesion strength on glass (kgf/cm^2)	20.23±1.24	42.76±2.73
Young's module (GPa)	9.5	265.8

[a]TGA test was performed in nitrogen ambient at the heating rate of 10°C/min.
[b]Specimens thickness for transparency measurement was about 100μm.
[c]it presents the amount of moisture penetrating through resin film sample in 24 hrs.
[d]moisture absorption was measured as following: the resin film samples were dipped into deionized H_2O for 24 hrs. The samples were then wiped dry completely and a high precision balance was employed to measure the absorbed H_2O.

TABLE II
THERMAL PROPERTIES OF NANOCOMPOSITE RESIN CURED AT DIFFERENT ENERGY DENSITY

	T_d	Tg
3.0 J/cm^2	176.7	106.8
9.5 J/cm^2	173.3	106.2

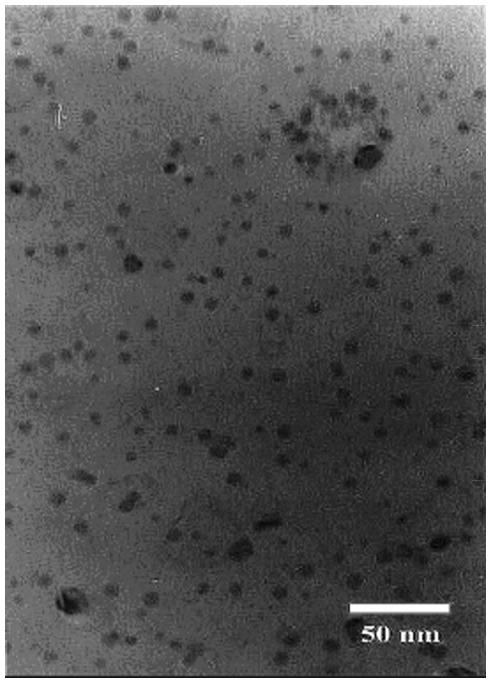

Fig. 2. TEM micrograph of the photo-curable *co*-polyacrylate/silica nanocomposite resin.

the source of UV irradiation comes from an array of five 8-W discharge tubes emitting UV light in the wavelength range of 315 to 340 nm with 120-mJ/cm^2 exposure energy density. The total UV exposure energy density was set at two levels, 3.0 and 9.5 J/cm^2, in order to investigate the influence of curing condition on breakdown voltage and leaking current density of resin films. In conventional OLEDs packaging process, the

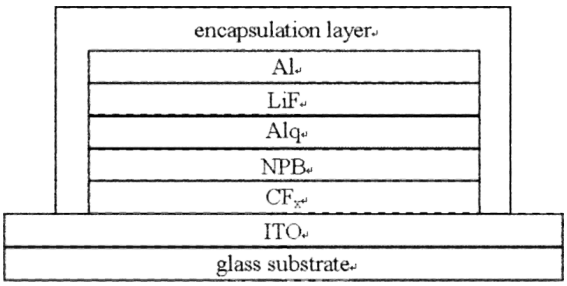

Fig. 3. Schematic representation of directly encapsulated OLED.

sealing resin is cured by a two-step process consisting of UV curing and post thermal baking at a temperature below its T_g. However, since the light-emitting material of OLEDs is sensitive to heat, the subsequent curing of resins by UV irradiation only is desired. Hence, the electrical properties of resins cured without post thermal baking were also evaluated. After curing, the top electrode consisting of layers of Ti, Cu, and Au was deposited on the resins to form the metal–insulator–metal (MIM) structure. The MIM samples were then sent to an HP 4156B semiconductor parameter analyzer operating in the range of 0 to 50 V for electrical property characterization.

C. Device Structure and Encapsulation Process

The OLEDs were fabricated on an ITO glass substrate (sheet resistance $= 20\ \Omega/\text{sq.}$) precleaned in a sequence of anhydrous acetone, deionized water, and methanol in an ultrasonic bath. The oligomer and nanocomposite resins were, respectively, coated on the OLEDs for encapsulation using a doctor-blade coating system, and its average thickness was about $68 \pm 1\ \mu$m for oligomer and $61 \pm 1\ \mu$m for nanocomposite resin as measured by a surface profiler (Veeco Dektak^3 ST). The encapsulated and the luminance area on the OLEDs was about 30×30 mm^2 and 3×3 mm^2, respectively, and the structure of the encapsulated OLEDs was schematically illustrated in Fig. 3. After the encapsulation, the OLED samples were sent to a UV oven and cured according to predetermined curing condition.

D. Luminance and Lifetime Measurement of Directly Encapsulated OLEDs

The changes of luminance, driving voltage, and lifetime of OLED samples were evaluated at varied current densities. The system for the luminance/lifetime measurement of OLEDs consists of a Photo Research PR50 spectrophotometer in conjunction to a Keithley 2400 source meter. The lifetime system is mounted in a glove box in which the moisture and oxygen concentrations are less than 1.0 and 4.0 ppm, respectively. A constant current attachment and photodiode arrays were adopted to monitor the luminance change of OLEDs as a function of time. The system was linked to a LabVIEW program for data acquisition and recording.

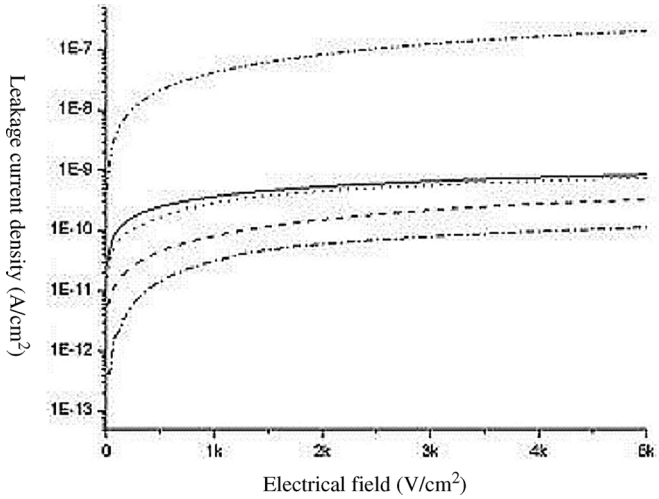

Fig. 4. Plot of leakage current density versus electrical field for oligomer subjected to UV curing at 3.0 J/cm^2 and post baking (———), oligomer subjected to UV curing at 3.0 J/cm^2 (– – ––), nanocomposite resin subjected to UV curing at 3.0 J/cm^2 and post baking (· · · · · · · ·), nanocomposite resin subjected to UV curing at 3.0 J/cm^2 (– · –), and nanocomposite resin subjected to UV curing at 9.5 J/cm^2 (– · ·–).

III. Results and Discussion

A. Electrical Properties of Photocured Resin Films

Since the encapsulated resin was directly coated on the cathode of OLED samples, electrical insulation would be an important property of practical applications. Fig. 4 presents the plot of leakage current density versus electrical field for oligomer and nanocomposite resin films subjected to different curing conditions. It can be seen that, with appropriate UV curing condition, the leakage current densities of cured films are below 10^{-9} A/cm^2, and no electrical breakdown was observed in the voltage range of test. The nanocomposite film exhibited better electrical properties in comparison with the oligomer subjected to the same curing condition. This evidences that the generation of inorganic nano-sized fillers clad in ethoxy groups [20] into oligomer to form the nanocomposite resin indeed benefits the insulation properties. Furthermore, regardless of resin type, post thermal baking in fact deteriorated the electrical properties of resins. Since the resins prepared in this work were cured via radical photo-polymerization, post thermal baking hence might be deleted from the curing procedures.

It is known that the condition of UV curing affects the properties of polymeric and polymer composite resins such as mechanical properties [23], [24], gas permeability [25], flame retardation [26], [27], etc. Fig. 4 also indicates that UV curing energy density has important effect on the electrical properties of resins. Deterioration of leakage current density was observed in the nanocomposite film cured at high exposure energy density of 9.5 J/cm^2. According to the result of viscoelastic measurement shown in Fig. 5, at the temperature 50 °C higher than T_g, the storage modules (E') of nanocomposite resin cured at 3.0 J/cm^2 is 217.2 MPa and that of resin cured at 9.5J/cm^2 is 181.2 MPa. Decrease of modules at the rubbery plateau of nanocomposite resin indicates that exposure energy density

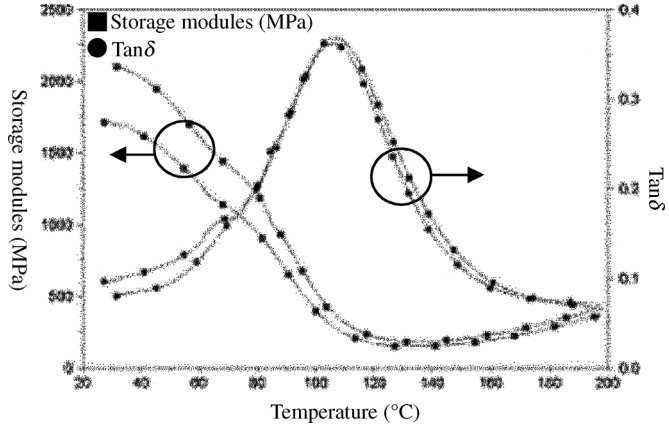

Fig. 5. DMA spectrum of *co*-polyacrylate/silica nanocomposite films cured at 3.0 J/cm^2 ($- \cdot -$) and 9.5 J/cm^2 ($- \cdot \cdot -$).

9.5J/cm^2 is too high for photo-polymerization due to the degradation of crosslinking chains. Another evidence for this comes from the changes of chemical structure of resins subjected to photocuring. Fig. 6(a) and (b) presents the Fourier transform infrared (FTIR) spectra of the oligomer and nanocomposite resins cured at 3.0 and 9.5 J/cm^2. As shown in Fig. 6(a), with the increase of UV curing energy the spectra for cured oligomer show continuous rise of absorbance of functional bands such as $-$OH around 3500 cm^{-1}, stretching C $=$ O near 1730 cm^{-1}, and asymmetric stretching C$-$O at 1180 cm^{-1}. This indicates the occurrence of photo-oxidation in oligomer resulted from UV irradiation [28], [29]. However, an FTIR spectra comparison between oligomer and nanocomposite resins shows that less photo-oxidation occurs in nanocomposite resin. Further FTIR and thermal analyses shown in Table II attributed the suppression of photo-oxidation to the scission of short polymeric segments in nanocomposite resin cured at 9.5 J/cm^2. It is known that excessive UV exposure induces photo-oxidation or photo-degradation and subsequently generates radicals, traps of electrons, or ions diffusion in polymeric coils [30]. When an external electrical field is applied, the presence of ionic/radical polymeric segments derived from radiation-induced polymer degradation would become the permeating paths and lead to the leakage of current [31]. Hence, the nanocomposite resin film cured at 9.5 J/cm^2 comprises higher leakage current density than that at 3.0 J/cm^2.

Since there is no report on the electrical properties of photocured polyacrylate resins for OLED encapsulation, it is difficult to determine that the leakage current in the range of several picoamperes would be significant for polyacrylate nanocomposite resin serving as the device packaging material. Nevertheless, the photocured nanocomposite resin in this work did comprise satisfactory electrical properties in comparison with oligomer for OLED encapsulation.

B. Device Characteristics and Luminance Property

Fig. 7(a) and (b), respectively, show the plots of current density versus driving voltage of the bare and encapsulated OLEDs. Luminance and driving voltage of OLED encapsulated with different types of resins operating at 20 mA/cm^2 are summarized in Table III. It shows that the relationships of current density

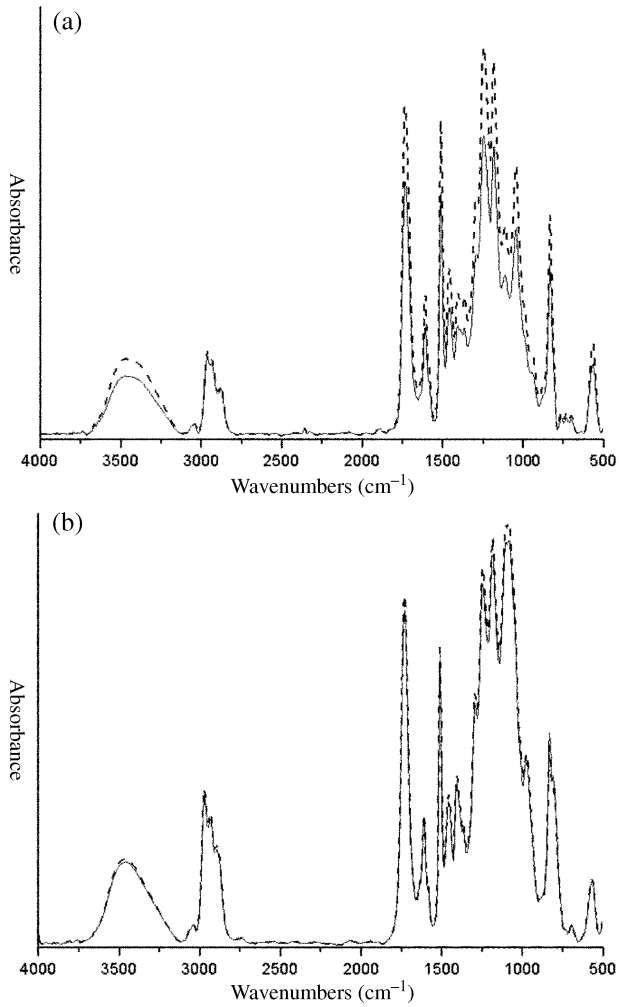

Fig. 6. FTIR spectra of (a) the oligomer and (b) the *co*-polyacrylate/silica nanocomposite resins cured at 3.0 (———) and 9.5 J/cm^2 ($- - - -$).

versus driving voltage are quite similar for OLEDs encapsulated by various resins cured at 3.0 J/cm^2. As shown in Table III, at 20 mA/cm^2 the driving voltage of the oligomer-encapsulated OLED is about 0.6 V higher than that of bare OLED, and the voltage difference between bare and nanocomposite-encapsulated OLEDs is less than 0.1 V. For the OLEDs encapsulated by the nanocomposite resin cured at 3.0 and 9.5 J/cm^2, the voltage difference is approximate 0.2 V at 20 mA/cm^2. According to the light-emitting principle of OLEDs, the electrons are injected from the top cathode then reach the electron transport layer (ETL) for subsequent combination with holes in emitting material layer (EML). If the OLEDs were encapsulated by resins with high leakage current densities, the electrons might also permeate into the encapsulated layer so that more electrons have to be generated to reach the threshold concentration by increasing driving voltage. The aforementioned electrical measurement indicated that the nanocomposite film possessed better insulation properties than that of oligomer and higher curing energy density resulted in higher leakage current density. Fig. 4 shows that the leakage current densities of oligomer cured at 3.0 J/cm^2, nanocomposite cured at 3.0 J/cm^2, and nanocomposite cured at 9.5 J/cm^2 are 5.26, 1.94, and 2×10^{-9} A/cm^2, respectively. Subsequent OLED characterization (see data shown in Table III) ev-

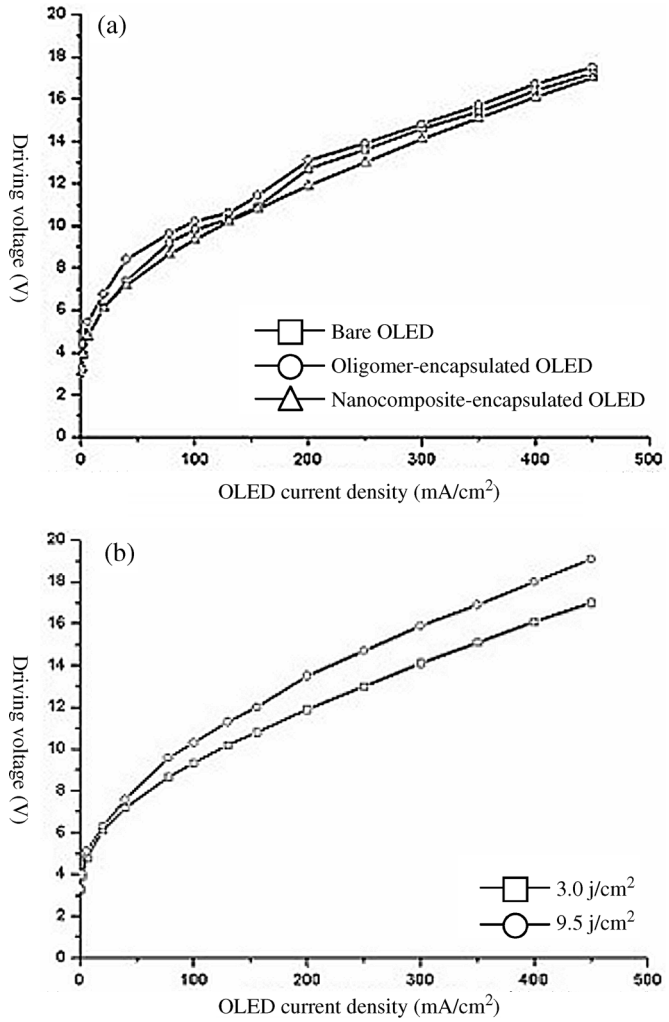

Fig. 7. Driving voltage versus current density of (a) bare OLED and OLEDs encapsulated by oligomer and nanocomposite resins UV cured at 3.0 J/cm^2. (b) OLED encapsulated by nanocomposite UV cured at 3.0 and 9.5 J/cm^2.

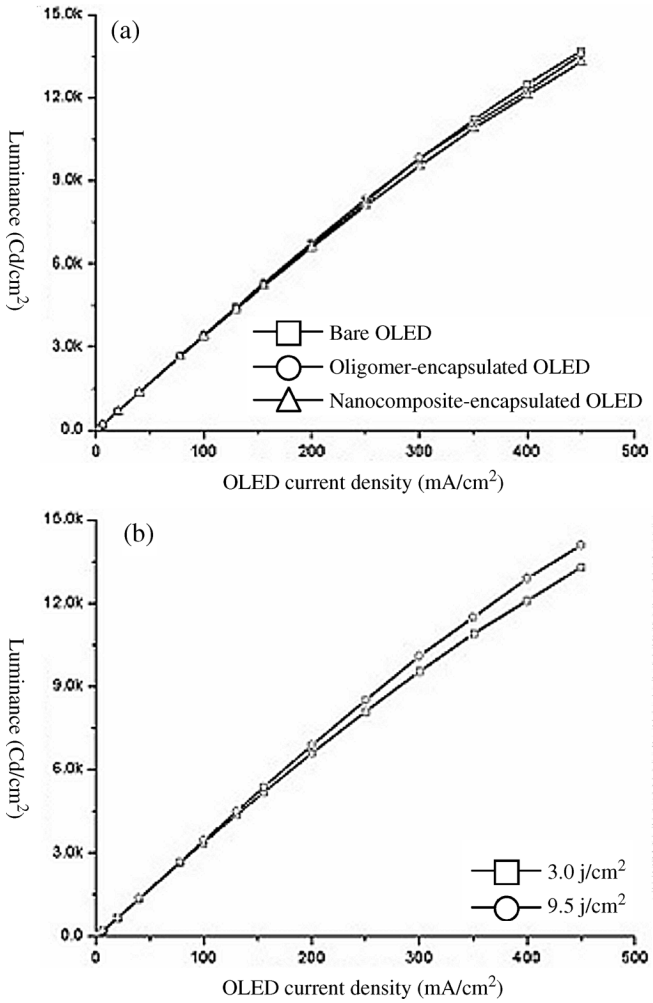

Fig. 8. Luminance versus current density of (a) bare OLED and OLEDs encapsulated by oligomer and nanocomposite resins UV cured at 3.0 J/cm^2. (b) OLED encapsulated by nanocomposite UV cured at 3.0 and 9.5 J/cm^2.

TABLE III
CHARACTERISTICS OF BARE/ENCAPSULATED OLEDS AT 20 mA/cm^2

Efficiency / Type of OLEDs	Luminance (cd/m^2)	Driving voltage (V)
Bare	673.0	6.15
Encapsulated with oligomer cured at 3.0 J/cm^2	669.4	6.77
Encapsulated with nanocomposite cured at 3.0 J/cm^2	666.0	6.09
Encapsulated with nanocomposite cured at 9.5 J/cm^2	664.0	6.29

idenced that the leakage current densities of encapsulated resin layers indeed affect the driving voltage of OLEDs.

Fig. 8(a) and (b) show the luminance versus current density of the bare and encapsulated OLEDs. With the increase of current density, there is no obvious difference on the luminance of the bare and encapsulated OLEDs up to 100 mA/cm^2 [8]. At 20 mA/cm^2, the differential ratio of luminance between the bare and the oligomer-encapsulated OLED is about 0.53%, and that between the bare and the nanocomposite-encapsulated OLED is about 1.04%. In addition, differential ratio of luminance is about 1.34% between the bare and the nanocomposite-encapsulated OLED cured at 9.5 J/cm^2. In the fabrication of top-emitting OLEDs, the radiation damages resulted from the ITO deposition would dominate the imperfections in organic layers stack [32]. Similarly, the photo-polymerization would possibly induce the damages in OLEDs. However, no obvious luminance change was observed in the OLEDs encapsulated by various resins. This implies that photo-polymerization process carried out in this work does not damage the light-emitting materials (Alq) and the cathode metal (LiF:Al). This part of study also evidences that the highly transparent nanocomposite resin is an alternative with great potential for direct encapsulation of OLEDs.

C. Lifetime Measurement of OLEDs

Fig. 9(a) presents the normalized luminance versus times for bare and encapsulated OLEDs operated at 20 mA/cm^2. In this paper, we define the lifetime of OLEDs as the time span for normalized luminance of the device drops to 80% (t_{80}). For the bare OLED, $t_{80} = 16.83$ h. For the oligomer- and nanocomposite-encapsulated OLEDs, the t_{80} are 178.17 and 350.17 h, respectively. The degradation mechanisms of OLEDs include 1) deterioration of light-emitting layer (EML), 2) material interdiffusion, 3) oxidation of active cathode metal, and 4) lack of hermetic capability of sealing material. In our experiment, the degradation phenomena related to mechanisms 1) to 3)

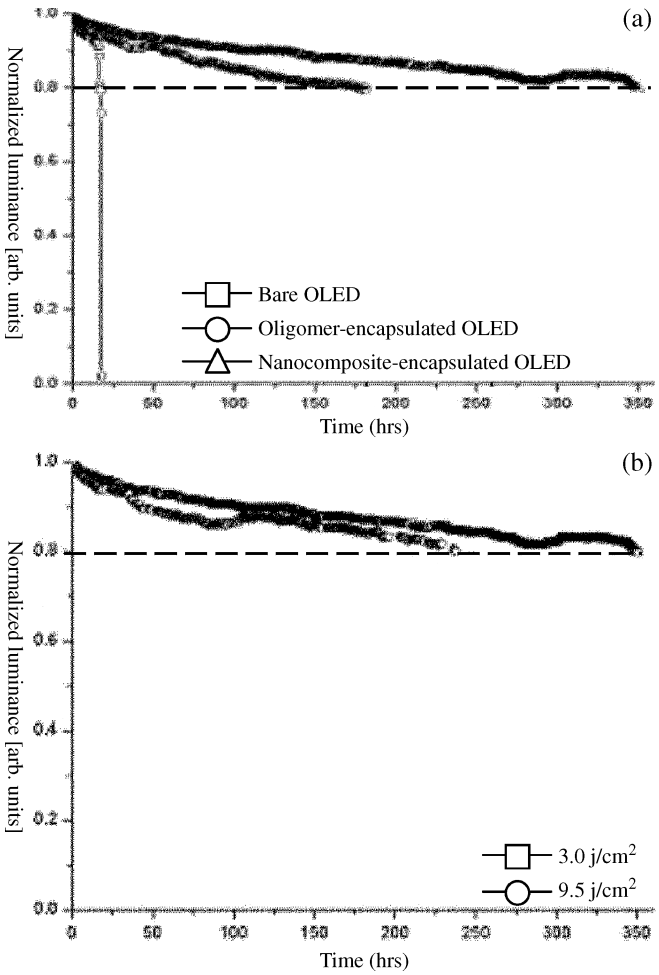

Fig. 9. Lifetime measurement of (a) the bare and encapsulated OLEDs and (b) nanocomposite-encapsulated OLEDs UV cured at 3.0 J/cm^2 and 9.5 J/cm^2 at current density of 20 mA/cm^2.

were either excluded or not observed. The improved lifetime of OLED encapsulated by nanocomposite resin presented in Fig. 9 hence attributed to the nano-sized silica particles embedded in resin matrix which effectively serving as the barrier of moisture permeation. In addition, improved adhesion strength of nanocomposite layer also benefits the device lifetime by inhibiting the penetration of harmful elements into the OLEDs via the resin/glass substrate interface. This is resulted from the fact that, during UV curing, the stable radicals derived from the UV irradiation would react with O_2 in the ambient to form peroxyl radicals. These radicals would further abstract hydrogen from polymer matrix [30] so as to increase the adhesion strength by forming hydrogen bonds at the resin/glass interface. High adhesion strength and low moisture permeability of nanocomposite encapsulation layer hence provide a better lifetime property for encapsulated OLED.

This work also found that UV curing energy density affects the lifetime of encapsulated OLEDs. As shown in Fig. 9(b), the t_{80} is equal to 237.67 h for OLED encapsulated by nanocomposite cured at 9.5 J/cm^2. McCaig *et al.* [25] reported that the deterioration of gas permeability of polyacrylate portion in the nanocomposite resin is deduced from its photo-degradation. The adhesion strength would also be degraded by the fracture of crosslinking chains in polymer matrix as revealed by DMA and FTIR analyses. The deterioration of moisture permeability and adhesion strength would reduce the hermetic sealing ability of nanocomposite resin so that the lifetime of OLED cured at high UV energy density was reduced.

IV. Conclusion

This work utilized self-synthesized *co*-polyacrylate/silica nanocomposite resin with high transparency and lower moisture permeability for direct encapsulation of OLEDs. Electrical property characterization indicated that the nanocomposite resin film subjected to appropriate UV curing treatment possesses lower leakage current density ($<10^{-9}$ A/cm^2). This provided the nanocomposite-encapsulated OLED a lower driving voltage in comparison with that for the oligomer-encapsulated OLED. The luminance measurement shows that the encapsulated resin type has very little effect on the device performance and the UV photo-polymerization carried out in this work does not damage the constitution of OLED. The lifetime of nanocomposite-encapsulated OLED was nearly twice as long as that of oligomer-encapsulated OLED. The improved device lifetime was attributed to low permeability property resulted from the formation of nanometer-sized silica particles in resin and high adhesion strength property resulted from the formation of hydrogen bonds at the resin/glass substrate interface. The experimental results evidenced that the is a thinner, lighter OLED packaging structure could be achieved via the method of direct encapsulation utilizing nanocomposite resin with optimum physical properties.

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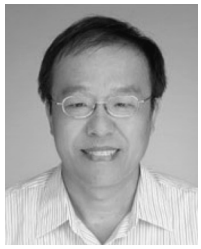

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