Integrating sphere charge coupled device-based measurement method for organic light-emitting devices

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(Received 10 February 2003; accepted 6 April 2003)

An integrating sphere charge coupled device (CCD)-based measurement system has been developed to accurately characterize the optoelectronic performance of organic polymer light-emitting devices (PLEDs). By theoretically analyzing a previously developed lens-coupled method and comparing it with the integrating sphere CCD-based method, we have found that the integrating sphere-based measurement method provides more stable reliable optical data in comparison with the lens-coupled measurement method. In addition, we demonstrate that inappropriate calibration of the PLED measurement system can greatly exaggerate device performance. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1581394]

To accurately determine the photoluminescence (PL) quantum efficiency of organic thin films, an integrating sphere has been used with a charge coupled device (CCD) spectrometer[1] or a scanning monochromator with a photomultiplier as a detector.[2] Both methods consider the photon energy at each wavelength to avoid any error in calculation that can be caused by using the average photon energy regardless of the broad emission spectrum of organic thin films. However, for electroluminescence (EL) external quantum efficiency measurement of organic light-emitting devices (OLEDs), a luminance meter was used to first measure the normal-direction luminance of the device, and then the EL spectrum was measured separately.[2] Since a luminance meter is usually calibrated with a large Lambertian light source, it may cause unintentional errors when the same optical coupling method used for calibration is applied to measurement of the optical properties of small OLEDs. Although the error can be reduced if a specially designed luminance probe is used to narrow the actual measurement area of the light source, the size and location of OLEDs with respect to the probe end still need to be carefully selected. In our laboratory, to avoid unintentional measurement errors due to the rather small size of the OLEDs and to accurately characterize the optical properties of the device, we developed an integrating sphere-based measurement system, in which a photodiode was initially used.[3] In this Note, we report organic polymer light-emitting device (PLED) data measured with an improved measurement system based on an integrating sphere and a CCD spectrometer that provides stable, accurate radiometric and photometric data such as the luminous flux, luminance, and external EL quantum efficiency of OLEDs. We also compare lens-coupled and integrating sphere-based measurement methods and discuss a possible unintentional error that can be caused by inappropriate calibration of the measurement system.

Figure 1(a) shows the integrating sphere-based measurement system (Meas. A) used in this study, where a CCD spectrometer is used as a detector. In this case, the fabricated PLED and the optical fiber connected to the CCD spectrometer are mounted on input and detector ports of the integrating sphere, respectively. As shown in Fig. 1(a), the whole system was calibrated with an irradiance standard lamp whose irradiance spectral distribution was provided by the manufacturer. Calibration will produce a set of conversion curves which convert the CCD response into luminous flux and emitted photon density spectral distributions. Details of the procedures will be described. Figure 1(b) shows a lens-coupled measurement system (Meas. B) previously developed by our group to characterize PLED performance in which a convex lens is located between the optical fiber connected to the CCD spectrometer and the PLED, at two focal lengths from each side. The calibration method of the CCD spectrometer for the lens-coupled measurement system is also shown in Fig. 1(b). Initially the radiance spectral distribution of the light source was used to produce the conversion curves.[4] However, since it is the total irradiance flux spectral distribution that actually couples the light source to the optical system, the radiance-based calibration method can cause unintentional exaggerated device performance.[5] Therefore, in this Note, we provide a modified calibration method for the lens-coupled measurement system to avoid any calculation errors, and it is described next. In addition, by analyzing the measured optoelectronic PLED data based on two different calibration methods, we show how inappropriate calibration can affect device performance.

As shown in Fig. 1(a), the PLED to be tested is mounted on the input port of the integrating sphere. In this method, we can only measure the CCD spectral response (counts/s nm) that corresponds to the total amount of light coming into the integrating sphere from the mounted PLED. However, in order to obtain the radiant flux spectral distribution (W/nm) from this CCD spectral response, we need to first know the relationship between them, which can be expressed by appropriate conversion curves after the measurement system is

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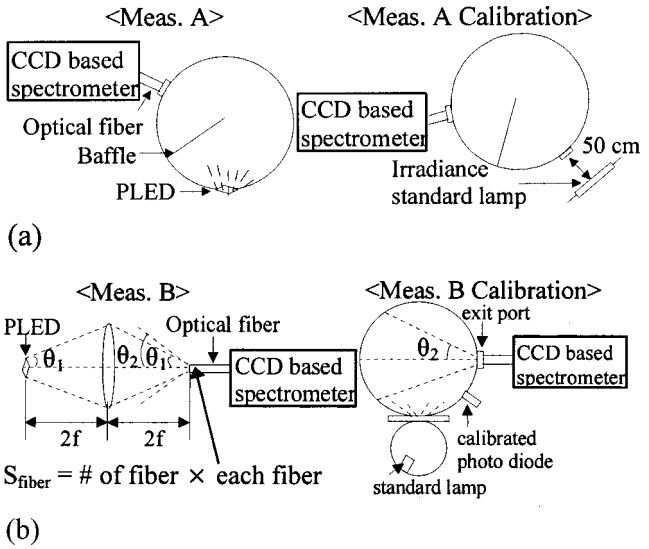

FIG. 1. Schematics of (a) an integrating sphere-based measurement system (Meas. A) and its calibration method and (b) a lens-coupled measurement system (Meas. B) and its calibration method. A CCD-based spectrometer was used as a detector for both methods. An irradiance standard lamp and a Labsphere uniform light source (Ref. 4) are used as a calibration standard light source for Meas. A and Meas. B, respectively.

calibrated with a standard light source. Now details of the calibration procedure are described. As shown in Fig. 1(a), a standard irradiance lamp is used, whose irradiance spectral distribution ($W/nm\,cm^2$) 50 cm from the lamp is provided by the lamp's manufacturer. These data are denoted as lamp irradiance in Fig. 2(a). To calibrate the whole system, we put the standard lamp 50 cm from the input port of the integrating sphere as shown in Fig. 1(a). The photopic eye response curve, also included in Fig. 2(a), converts radiometric data to photometric data, and weights the radiant flux to match the responsivity of the human eye over various wavelengths.[6] Since the irradiance of the standard lamp is radiant flux per unit area, we can produce the total radiant flux through the input port of the integrating sphere by multiplying the area of input port (1.27 cm diam) by the irradiance response, which is denoted as radiant flux in Fig. 2(b). Figure 2(b) also includes the luminous flux from the standard lamp through the input port obtained by multiplying the calculated radiant flux by the photopic eye response curve. Then, we measure the CCD spectral response for a standard lamp, which is shown in Fig. 2(c). When we divide the radiant flux and luminous flux by the CCD spectral response, conversion curves can be obtained, shown in Fig. 2(d), which change the CCD spectral response to the radiometric and photometric data. In Fig. 2(d), the radiant flux is converted to the photon number by considering the photon energy at each wavelength. Therefore, by multiplying the conversion curves by the CCD spectral response measured for PLED light emission, we can directly calculate the desired photometric and radiometric spectral distribution of the device.

A similar conversion procedure for the lens-coupled measurement system was used.[4] The initial approach to produce conversion curves was radiance-based calibration of the measurement system, where the radiance spectral distribution ($W/sr\,nm\,m^2$) of the light source used was used as a fundamental optical quantity. However, during the postdata

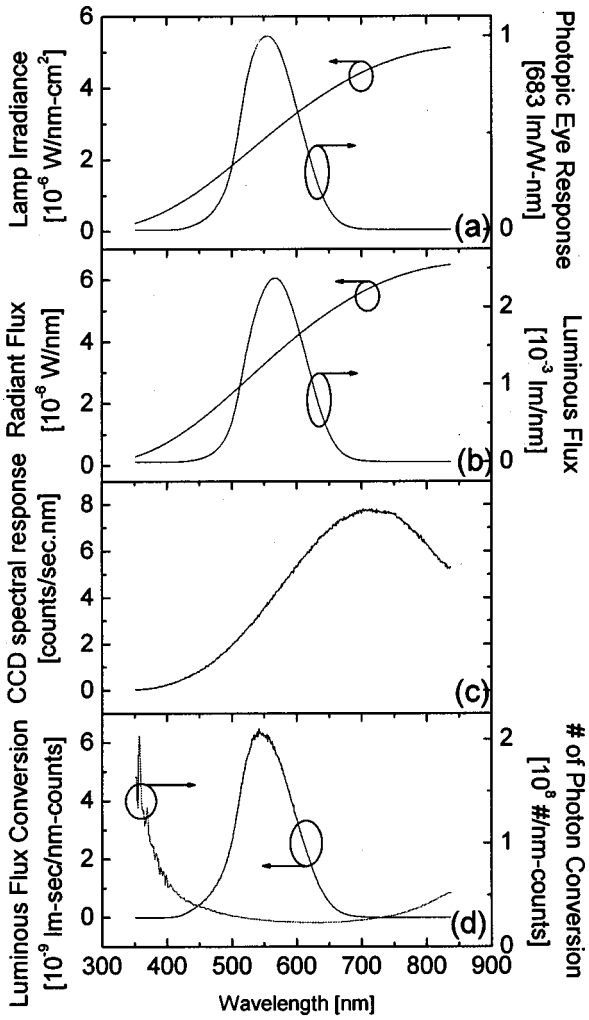

FIG. 2. Procedure for calibration of the integrating sphere-based measurement system: (a) irradiance of the standard lamp and photopic eye response, (b) optical power and luminous flux spectral distribution calculated by considering the input port size (1.27 cm diam) and photopic eye response, (c) measured CCD raw response, and (d) conversion curves extracted from (b) and (c).

process in which the lens effect of the PLED measurement system was considered, the initial method can lead to unintentional exaggerated device performance[5] since the limited acceptance angles of the optical fiber and the lens optic can be used inappropriately in the radiance-based calibration method. Therefore, we further analyzed our previous method by considering the acceptance angle ($\theta_2 \sim 12.7°$) and area ($S_{\text{fiber}} \sim 4.27\times10^{-6}\ m^2$) of the optical fiber bundle used (136 fibers with 200 μm diam) shown in Fig. 1(b). This improved method is summarized as follows, where the total irradiance flux spectral distribution from the light source to the optical fiber was used throughout the whole calibration procedure (irradiance flux-based calibration method).

(a) Calculate the total irradiance flux spectral distribution (E_{source}) coupled to the optical fiber bundle by considering the acceptance angle and area of the optical fiber bundle used.

$$E_{\text{source}} = R_{\text{source}} \times \pi \times (\sin\theta_2)^2 \times S_{\text{fiber}}, \quad (1)$$

where R_{source} is the radiance spectral distribution of the light source.[7]

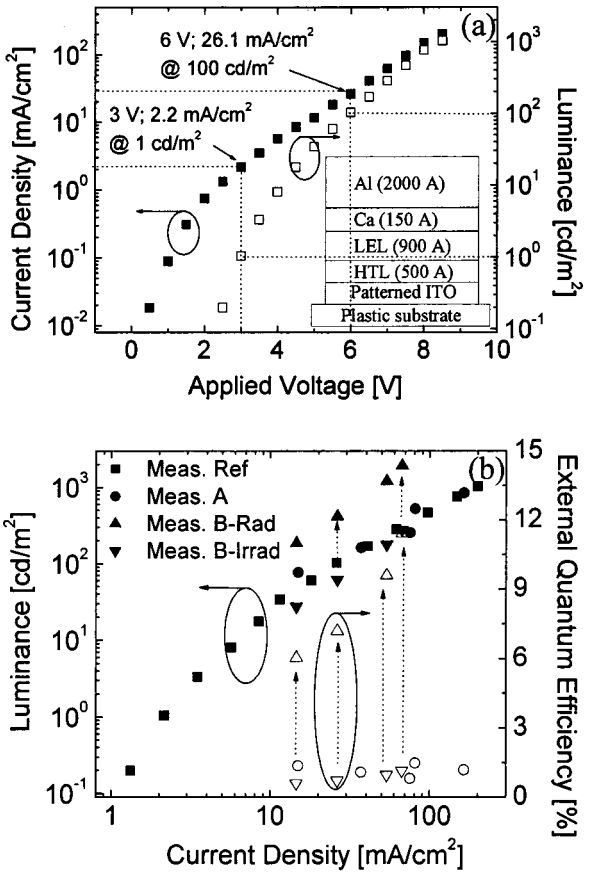

FIG. 3. (a) Current density and luminance vs voltage applied for PLEDs fabricated on flexible plastic substrates, which are measured with a measurement system based on an integrating sphere and a photodetector. The structure of the PLED used in this study is also included. (b) Luminance and external quantum efficiency vs current density characteristics of different measurement methods, where Meas. B-Rad, and Meas. B-Irrad represent results from a lens-coupled measurement method with radiance-and irradiance flux-based calibrations, respectively.

(b) Measure the CCD spectral response that corresponds to the radiance spectral distribution (or the calculated total irradiance flux spectral distribution) of the light source.

(c) Extract conversion curves by dividing the CCD spectral response (b) by the total irradiance flux spectral distribution (a).

(d) Measure the CCD spectral response for PLED light emission with the lens-coupled measurement system.

(e) Calculate the total irradiance flux spectral distribution (E_{PLED}) that corresponds to PLED light emission by multiplying the measured CCD spectral response (d) by the conversion curves extracted (c).

(f) Calculate the PLED radiance spectral distribution (R_{PLED}) by considering the acceptance angle of the lens-coupled measurement system, where the acceptance angle [$\theta_1 \sim 11.3°$ in Fig. 1(b)] defined by the lens determines the acceptance angle of the whole measurement system since $\theta_1 < \theta_2$.[7]

$$R_{\mathrm{PLED}} = \frac{E_{\mathrm{PLED}}}{\pi \times (\sin \theta_1)^2 \times S_{\mathrm{fiber}}}. \tag{2}$$

(g) Calculate the photometric and radiometric data of PLED from the R_{PLED} obtained.

The effects of these two different calibration methods on PLED opto electronic performance will be discussed further by analyzing the PLED performance data next.

To evaluate two measurement systems and two calibration methods for the lens-coupled measurement system, we measured and compared the PLED optoelectronic performance. As a reference, we measured the PLED with a previously developed measurement system (Meas. Ref).[3] Figure 3(a) shows luminance–current density–voltage applied characteristics of the fabricated PLED. The PLEDs used in this study were fabricated on the plastic substrates shown in the inset of Fig. 3(a). The plastic substrates were cleaned in an ultrasonic bath of isopropanol before polymer deposition. Then, a hole transport layer (HTL) (~500 Å) and a light-emissive layer (LEL) (~900 Å) were consecutively spin coated on. Poly (9,9-dioctylfluorene-co-*N*, *N*′-di(phenyl)-*N*, *N*′ di (3-carboxyphenyl) benzidine) (BFA) and red emissive poly (fluorene) copolymer were used in DMF and xylene solvents for the HTL and LEL polymers, respectively. A calcium (~150 Å) and aluminum (~2000 Å) bilayer cathode was thermally evaporated without interruption through shadow masks under high vacuum ($\sim 10^{-6}$ Torr) at a rate of 1 and 5 Å/s, respectively. All device fabrication and measurement were performed under ambient conditions. Turn-on voltage and current density of 3 V and 2.2 mA/cm^2, respectively, are obtained, which are defined at luminance of 1 cd/m^2. At 100 cd/m^2, the driving voltage and current density are 6 V and 26.1 mA/cm^2, respectively. The emission/power efficiencies of the device are ~0.4 cd/A and ~0.2 lm/W, respectively, but are not shown in this Note.

Figure 3(b) shows the luminance and external quantum efficiencies versus the current density characteristics of the fabricated PLEDs, which were measured with a different system and using different calibration conversion curves. When the integrating sphere is used with a photodiode (Meas. Ref) and with a CCD spectrometer (Meas. A), the measured results (squares and circles) show consistent device performance. When the lens-coupled method with the radiance-based calibration curves (Meas. B-Rad) is used, the results (triangles) show exaggerated device performance. This exaggeration occurs because of inappropriate calibration of the measurement system. However, when the irradiance flux-based calibration method is used (Meas. B-Irrad), the measurement results (inverted triangles) show good agreement with the results obtained from Meas. Ref and Meas. A, and only slight deviation. This observed deviation is related to the unit alignment between the light-emitting source, the lens, and the optical fiber connected to the CCD spectrometer during measurement system calibration and device measurement. In comparing the measurement results between Meas. B-Rad and Meas. B-Irrad, we conclude that inappropriate calibration can cause unintentional exaggeration of device performance by up to 10-fold. Therefore, when any optical measurement system is calibrated, appropriate optical data should be considered for accurate measurement. Also, based on our results, an integrating sphere-based measurement system shows more reliable consistent measurement results.

The authors would like to thank Shu-jen Lee for theoretical and technical discussions regarding measurement system calibration. This research was supported by a DoD-DARPA grant.

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