Bright and color-saturated quantum dot light-emitting diodes, new star for next generation displays and solid state lighting

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Abstract: The emergence of solution-processed semiconductor quantum dot (QD)-light emitting diodes (LEDs) has recently offered a great prospect for developing low-cost, efficient, bright, and large-area colorful displays compatible with flexible substrates. The band-edge electroluminescence (EL) of colloidal QDs of cadmium compounds, exhibits size-tunable spectral emission (450-760nm) and narrow bandwidth (FWHM~15-30nm), allowing for the design and fabrication of color-saturated red, green and blue (RGB) QD-LEDs with simple device configurations and high spectral purities that outperform those of liquid crystal displays and organic light-emitting diodes. Additionally, high fluorescence quantum yield and photochemical stability can be achieved by engineering the nanocrystal surfaces with wide-bandgap shells, which favors the efficiency and stability of QD-LEDs. We report in this conference of our recently developed red, orange, yellow, green, blue and white quantum dot light-emitting diodes with high brightness, high efficiency, saturated color on both indium tin oxides (ITO) coated glass and on flexible PET substrate.

Keywords: quantum dots, light-emitting diodes, flexibility

1 Introduction

Recent advances in the synthetic techniques for growing high-quality colloidal quantum dots (QDs) of semiconductor compounds, such as (Zn,Cd)(S,Se) and (In,Ga)(As,P), have given rise to a new family of solution-processable inorganic chromophores featuring size-tunable color, narrow emission bandwidth, high photoluminescence (PL) efficiency, unmatched photochemical stability, and structural robustness. These superior properties render them promising building blocks for the next-generation thin-film optoelectronics, which has spurred the widespread interest of developing QD-based light emitting-diodes, [1-9] hybrid photovoltaics, [10,11] nanoscale lasers, [12,13] etc.[14] We will report in this conference of our recently developed red, orange, yellow, green, blue and white quantum dot light-emitting diodes with high brightness, high efficiency, and saturated color on both ITO coated glass and on flexible

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PET substrates.

2 Experiments

2.1 Materials

The semiconductor nanocrystal QDs used in this study were configured with core-shell structures, with CdSe emissive cores and CdS/ZnS double shells for red, orange, yellow and green QDs, and CdS emissive cores and ZnS shells for blue QDs.[15] The emission color of the QDs was tuned by tailoring the core composition/size and controlling the shell structure following the nanocrystal synthetic route based on inorganic precursors and non-coordinating solvents. [8,9]

2.2 Device fabrication

The fabrication of the QD-LED device begins with a UV-ozone treatment of the ITO substrate to enrich the negatively charged oxygen on the ITO surface and, subsequently, increase the ITO work function.[16] The treatment also helps to enhance the wetting of the ITO surface by removing any residual organic carbon contaminants and modifying ITO surface polarity.[17] On the UV-ozone treated ITO surface we spun-coat PEDOT:PSS (~30 nm), which was followed by an oven-annealing at 120℃ for 10 min at atmosphere pressure. Next, the sample was transferred into a nitrogen glovebox system with controlled concentrations of oxygen (≤5 ppm) and water vapor (≤1 ppm), and the poly(N,N'-bis(4-butylphenyl)-N,N'-bis(phenyl)benzidine) (poly-TPD) hole transporting layer (HTL), ~45 nm in thickness, was spun cast on top of the PEDOT:PSS layer from the chlorobenzene solution and cured at 150℃ for 30 min on a hot plate. Subsequently, the emissive layer of Cd(S,Se)/ZnS nanocrystal QDs with different emission wavelength was spin-deposited over the surface of poly-TPD HTL from the toluene solution and baked on a hot plate at 80℃ for 30 min to form the active region of the QD-LED. The thickness of the QD-emissive layer has been precisely tailored, by varying the QD concentration in the solution and the spin speed of the cast deposition, to between 1-5 monolayers depending on the QD size (and therefore the color) to balance the maximum brightness and emission efficiency of the QD-LEDs, as reported elsewhere.[18] A ~30nm thick-Alq3 ETL was then thermally deposited, under a pressure of 3×10^{-7} torr, over the QD-active region. Finally, a Ca/Al (10 nm/150 nm) bi-layer cathode was thermally evaporated through a shadow mask without breaking the vacuum. The fabricated QD-LEDs have circular shapes, defined by the shadow mask, and each has a surface area of ~7 mm^2. It is noted that the Alq3 ETL was excluded from the blue QD-LED structure to suppress the residual organic emission for the maximum color saturation of the blue QD-LED output due to the low luminous efficacy of the blue light.

2.3 Device characterization

The current-voltage characterization of devices were performed with a Keithley 4200 semiconductor parameter analyzer, while an Ocean Optics UV-Vis spectrometer was used to study the LED output spectra. To determine the photometric brightness (cd/m^2) of the device, the light output was first measured with a calibrated Newport 1830-C power meter utilizing an 818SL silicon photodetector directed, at a fixed distance, toward the ITO side of the device. The

LED luminance (brightness) was then calculated from the known portion of the forward emission and the LED output spectra. Images of LED output were recorded with a Sony FWX700 FireWire color CCD camera. All the measurements were performed under ambient conditions at room temperature.

3 Results and discussion

3.1 Developing multicolored quantum dots LEDs

The multilayer structure of ITO/PEDOT:PSS (30 nm)/HTL (45 nm)/QDs (15~20 nm)/ETL (30 nm)/Ca (10 nm)/Al (150 nm) was employed in the fabrication of the QD-LEDs, with ITO/PEDOT:PSS as anode, poly-TPD spin-coated from chlorobenzene solution as HTL, QD layer spin-coated from the QDs toluene solution as emissive layer, Alq3 as ETL and Ca/Al as cathode. The device structures and energy levels of the materials involved in a red-emitting QD-LED are plotted in Fig. 1.

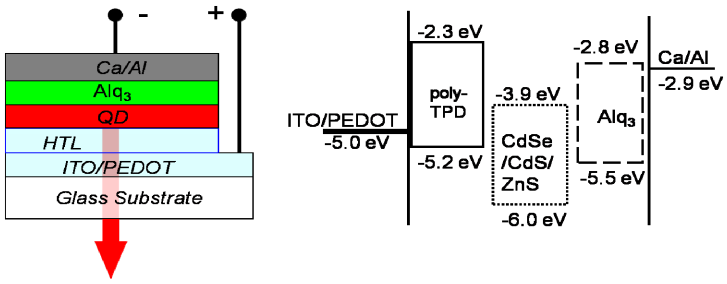

Fig. 1 Device structure and energy level of red quantum dots LED

Fig. 2 shows typical current and luminance curves as a function of applied voltage for the four color QD-LEDs. All the devices showed low turn-on voltages of 3-4 volts confirming the minimized barrier height for charge injection in the QD-LEDs. The maximum luminance and luminous efficiency reached 9064 cd/m^2 and 2.8 cd/A for the red device, 3200 cd/m^2 and 1.8 cd/A for the orange device, 4470 cd/m^2 and 1.3 cd/A for the yellow device, and 3700 cd/m^2 and 1.1 cd/A for the green device. At standard display brightness (100-500 cd/m^2), the four color QD-LEDs showed relatively stable and high luminous efficiencies of 2.68, 1.46, 1.30, and 1.06 cd/A at 100 cd/m^2 and went to 2.64, 1.14, 1.12, and 1.06 cd/A at 500 cd/m^2 for the red-, orange-, yellow-, and green-emitting devices, respectively. The power efficiencies of the four color QD-LEDs consistently reached the maxima at near turn-on and then decreased to 0.33-1.11 lm/W (green to red) at 100 cd/m^2 and to 0.27-0.78 lm/W at 500 cd/m^2.

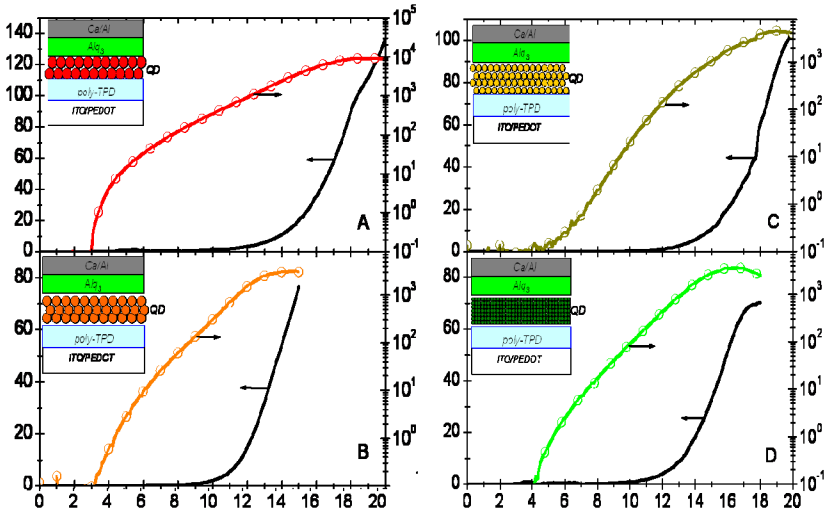

Fig. 2. I-L-V curves of red, orange, yellow and green QD-LEDs

EL spectra of the four-color QD-LEDs operated at a different brightness (voltage) are shown in Fig. 3. The EL emission of all the QDLEDs are desirable, with peaks centered at 619 nm, 595 nm, 576 nm and 525 nm, for the red-,orange-, yellow- and green-emitting devices, respectively. The FWHM of the EL peaks are 28 nm for the red and orange QD-LEDs, 35 nm for the yellow and 32 nm for the green devices, demonstrating the color-saturated light emission of the QD-LEDs. Furthermore, the EL spectra of all the QD-LEDs kept stable within the whole measured luminance (voltage) range from ca.10 cd/m^2 to the maximum luminance. There is no obvious contribution from the polymer HTL or organic ETL in the EL spectra of the QD-LEDs. The Commission Internationale de l'Enclairage (CIE) coordinates of the emitted lights of the QD-LEDs are (0.653, 0.324), (0.544, 0.418), (0.480, 0.485), and (0.202, 0.676) for red-, orange-, yellow-, and green-emitting devices, respectively.

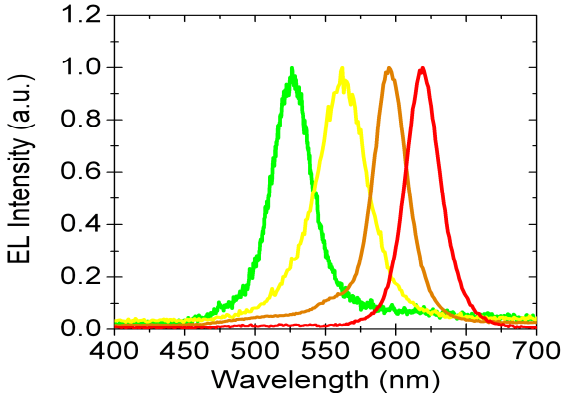

Fig. 3 EL spectra of red, orange, yellow and green QD-LEDs

3.2 Developing bright and color saturated blue QD-LED

To date only a few blue QD-LEDs have been demonstrated with maximum brightness of ~125cd/m^2 and full-width-at-half-maximum (FWHM) bandwidth ≥ 32nm. [7] In spite of the 'blue' peak wavelengths (440nm-478nm)

reported for those QD-LEDs, their emission spectra inevitably exhibited significant tails extending into the green and red region of the spectrum, the integrated intensities of which often contain ≥50% of the overall LED radiance power. Since the luminous efficacy of blue light is much lower than that of green light, a broadband, long-wavelength tail beyond the blue portion of the emission could make major contributions to the LED brightness, making the blue emission of QD-LED far from being saturated.

Herein we report the design and processing of a QD-LED whose brightness and blue purity far exceed the early reported values. The LED was configured with a multiple-layer structure employing structurally-engineered core/shell-CdS/ZnS QDs in the emissive region. At a low operation voltage of 5.5V, the device emitted spectrally-pure blue with a strikingly narrow FWHM-bandwidth of 19nm and a high brightness up to 1600 cd/m^2. The long-wavelength tail of the LED output was minimized to less than 5% of the total emission, leading to the highly-saturated blue emission from QD-LEDs. The result of our blue-LED study represents a significant improvement over the performance of existing blue QD-LEDs, and marks a further step toward the practical application of QD-LED technology in full-color display and solid state lighting. The I-L-V curves and the device structure as well as the current efficiency are showed in Fig. 4.

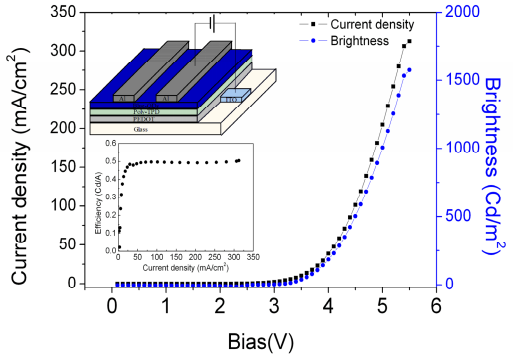

Fig. 4 I-L-V curves of blue QD-LED. Inset: the device structure and current efficiency of blue QD-LED.

Fig. 5 shows the EL spectrum of the device at the brightness of 1600 cd/m^2. The LED emission peak at 460 nm, with the measured FWHM-bandwidth is as narrow as 20nm. The spectral characteristic of the LED output resembles that of the PL emission of the QD solution except for a small shoulder in the UV region due to the residual emission from the poly-TBD (λpeak~410nm). There is no noticeable shift of the peak wavelength between the EL and PL emission. The integrated intensity of the broad, long-wavelength tail over the green and red spectral regions has been reduced to less than 5% of the total emission of the device, ensuring the spectral purity of blue light output from the QD-LED. The inset shows the photomicrographs of the LED surface taken at 100 cd/m^2 and 1600 cd/m^2, respectively. At high brightness, the LED image becomes whitish due to the saturation of the camera sensor pixels, while the periphery region glows blue as a result of the back scattering.

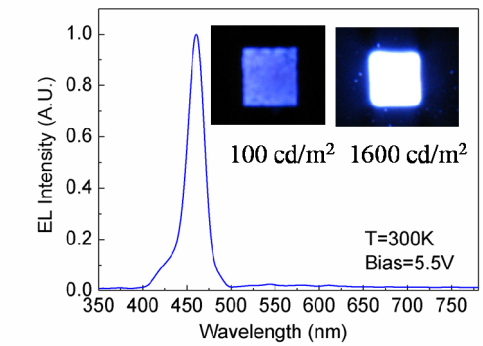

Fig. 5 EL spectrum of blue QD-LED. Inset: images of device operated at different voltage.

3.3 Developing stable binary Complementary white emission QD-LED

Among many of the potential applications of QD-LEDs, white-LED based solid- state-lighting has recently commanded much attention as a cost-competitive, energy-efficient alternative to conventional electrical lighting.[7,19-21] White QD-LEDs have been demonstrated with a host/guest system consisting of a blue-emitting polymer doped with red-emitting CdSe/ZnS core-shell QDs and a green emitting metal chelate complex Alq3. A fairly pure white light output with CIE coordinates of (0.30, 0.33) was achieved by carefully tuning the concentration ratio of the QD/polymer blend.[19] In another study, a mixed-monolayer of red, green, and blue emitting colloidal QDs was employed in the LED active region producing a broad spectral electroluminescence.[7] Precise tuning of the emission spectrum was accomplished by changing the ratio of different color QDs in the mixed layer, which led to the demonstration of white QD-LEDs that emitted at video brightness with the CIE coordinates of (0.35, 0.41) and high color rendering index. In both cases the multicolor emitters (QDs and/or organic molecules) were blended into nanocomposites constituting the active layers of devices, and the LED output color was controlled via varying the weight fraction of the constituents in the nanocomposites. The Förster energy transfer between the blue and green/red emitters in the nanocomposite was considered cruel for the color mixing process in the LED operation.[21]

We report in this letter a novel white QD-LED configuration wherein a layer of yellow-emitting CdSe/ZnS core/shell QDs was deposited over a thin film of blue-emitting polymer, Poly-TPD, creating a simple, bilayer EL device that generates binary complementary white light. By optimizing the thickness of the QD layer, we have achieved stable and bright white QD-LED operation over a wide range of bias. The onset voltage of the device was as low as 3.15 V, and the maximum brightness was measured to be 2600 cd/m^2, as shown in Fig. 6.

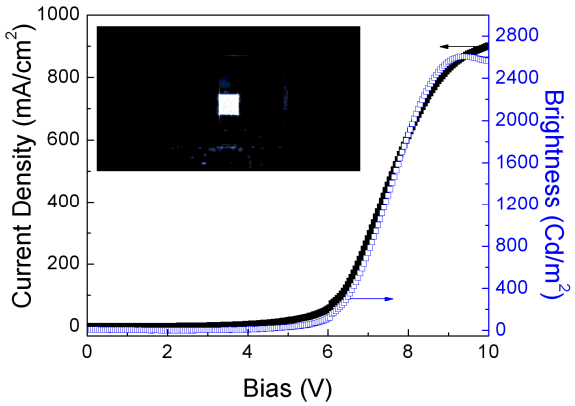

Fig. 6 I-L-V curves of white QD-LED. Inset: image of device worked at 2000 cd/m^2.

The EL spectra of the QD-LED were plotted in Fig. 7 for a set of bias voltage. It is evident from the spectra that spectral characteristic of the LED output exhibits little dependence over a significant portion of the voltage range (7-11V) of operation, resulting in a stable LED color with a large dynamic range of output brightness: When the bias voltage was varied from 7 V to 11 V, the luminescence of the QD-LED grew from 400 to 2600 cd/m^2, whereas the CIE coordinates of the LED color only shifts slightly from (0.315, 0.357) to (0.326, 0.374) in the SAE J578 white regime and remain close to the balanced white coordinates (0.333, 0.333).

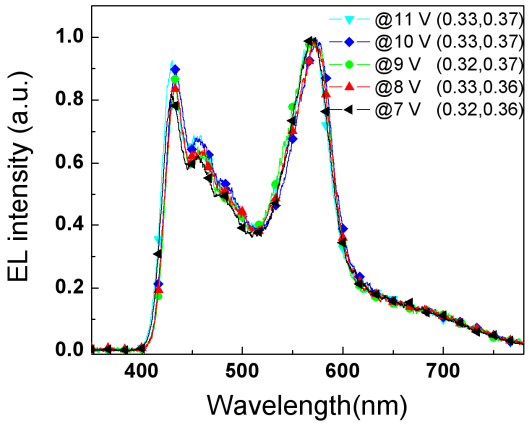

Fig. 7 EL spectra of white QD-LED operate at different voltage.

3.4 Developing flexible red, green and blue QD-LED

Until now, almost all the best QDs-based LEDs have been achieved on devices using rigid ITO coated glass substrate, which has lower permeation rate of moisture and oxygen, much smother surface and stronger adhesion. Typically, in these devices, each of the active layer and electrodes has a thickness of only about 100 nm, so the thickness and weight of the devices are dominated by the supporting substrate. On the other hand, glass substrates, however, do not allow exploitation of the intrinsic flexibility of LEDs to enable new light weight, rugged flexible displays produced by roll to roll manufacturing. Unlike glass, the plastic is usually more robust and compact, has lighter weight, and is more

cost effective. The use of the plastic substrates will also enable new product such as curved displays, smart cards, cellular phones, and all plastic electronics.[22-25]

Fig. 8(a) and (b) show a photograph and schematic configuration, respectively, of a bendable QD-LED. The chemical structures of polymer and molecular materials (PEDOT:PSS, poly-TPD, and Alq3) used in device are presented in Fig. 8c.

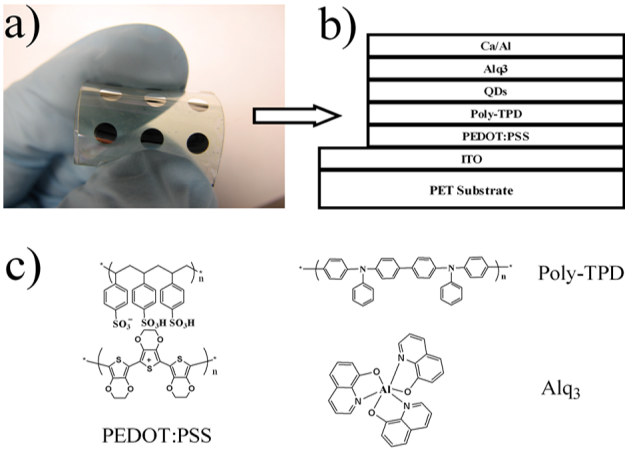

Fig.8 (a) Images of bended device; (b) Device structure of flexible QD-LED; (c) Molecule structure of materials.

The as-prepared QDs exhibited narrow size distribution and were single crystals free of structural defects, as manifested by the narrow-band PL emission of the QD solution and the well-resolved fringes in the high-resolution transmission electron microscopy (HR-TEM) images of individual nanocrystals (Fig. 9). The wavelengths of the PL maxima for red, green, and blue QDs dispersed in toluene are 628 nm, 535 nm, and 461 nm, respectively, and the full-width-at-half-maximum (FWHM) bandwidths of the PL signals vary between 20-30 nm. The average diameters of red, green, and blue dots were determined from their HR-TEM images to be 5.5, 3.5, and 3 nm, respectively.

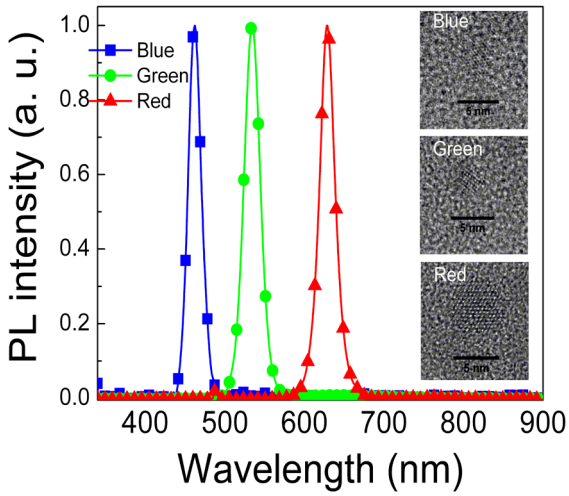

Fig. 9 PL spectra and TEM images of red, green and blue quantum dots.

The photographic images in Fig. 9 (a) display the output of red, green and blue (RGB) QD-LEDs working in relaxed and bent states, respectively. The close-up view of the surface emission from the relaxed RGB LED pixels operated at a brightness of ~200 cd/cm^2 reveals the spectral and luminous uniformity across the device emissive regions. Devices in the bent samples were imaged at the maximum luminance (~7000 cd/m^2 for red, ~3300 cd/m^2 for red, ~1400 cd/m^2 for red) to highlight the bright emission from the flexible QD-LEDs. At high brightness, the LED images become whitish due to the saturation of the camera sensor pixels, while the periphery regions glow in the respective colors as a result of the back scattering. The narrow-band electroluminescence emission of RGB QDs, recorded at the maximum LED brightness, is centered at 624nm, 535nm, and 462nm, respectively, with the corresponding FWHM bandwidths as 28nm, 30nm and 22nm, respectively. There is no noticeable difference between the output spectra for relaxed and bent QD-LEDs. The organic emission in the QD-LED output has been minimized via controlling the HTL and ETL layer thicknesses in the device configuration design. Fig.9 (b) shows the CIE chromaticity coordinates for the red (0.62, 0.32), green (0.21, 0.75) and blue (0.16, 0.06) colors of the RGB flexible QD-LEDs with respect to the National Television System Committee (NTSC) color triangle. It is evident from the CIE plot that the triangle formed by connecting the color coordinates of the QD-LEDs largely encloses the NTSC color triangle, suggesting the superior colormetric properties of the flexible QD-LEDs towards their potential applications in full-color and high-definition flexible displays.

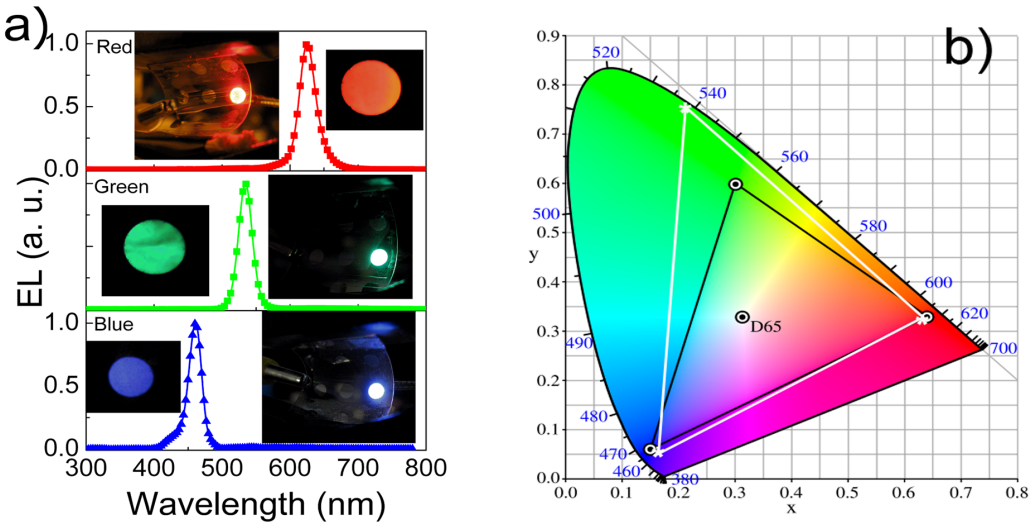

Fig. 10 (a) EL spectra and images at different stress state of red, green and blue flexible QD-LEDs; (b) CIE of quantum devices and NTSC standard.

Presented in Fig. 11 are a set of representative luminance-current-voltage (L-I-V) characteristic curves of the RGB flexible QD-LEDs, whereupon the luminous efficiencies (cd/A) have been calculated and also plotted as functions of bias in the insets of Fig. 11. Almost all the flexible devices investigated in the present study have low turn-on voltages (3-4 V), confirming the low barrier height for the QD-injection of charges in the LED operation. The maximum luminance and luminous efficiencies reach 7070 cd/m^2 and 2.57 cd/A for red devices, 3375 cd/m^2, and 2.32 cd/A for green devices, and 1408 cd/m^2 and 0.40 cd/A for blue devices, respectively, which are fairly comparable to the best performance hitherto

reported for the QD-LEDs fabricated over rigid glass substrates.

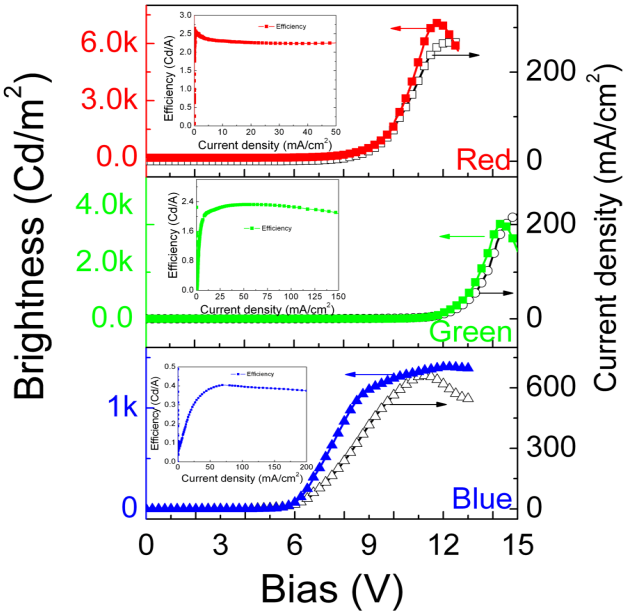

Fig. 11 I-L-V and current efficiency of flexible red, green and blue QD-LEDs

The flexibility of the PET-based QD-LEDs was examined by investigating the performance of the device in the bent state under mechanical stress at different bending radii. In the experiment, a thin-film sample of red QD-LEDs was bent with the device structure on the concave side of the film using the clamps of a sample holder, and light detection was performed through the PET/ITO substrate using a photodetector positioned at a constant distance from the bent device. The I-V and L-V characterization of the device bent into different states of curvature (Fig. 12(a) & (b)) suggest that the observed declination of QD-LED brightness with the bending radius can be correlated to the reduced current density of the device under the same voltage bias condition. The calculated LED luminous efficiency at different bending radii, as plotted in the inset of Fig. 12(b), offers some valuable insights to the EL emission of QDs in bent LED devices: the fact that the luminous efficiency remains largely the same at any bending radii larger than ~5mm suggests that the EL performance of the QD layer in the LED active region was not subject to any degradation under mechanical stress. Since the molecular components in the LED device are also highly compressive, this leaves the electrode degradation as the most likely failure mechanism in the flexed QD-LEDs. Such a postulation is supported by the observed critical radius (~5mm) for the current reduction in the flexed device (inset of Fig. 12(b)), which has been attributed to the onset of the cracking phenomenon in the ITO films in the previous studies on the polymer LEDs.[26] It is worth mentioning that the critical bending radius of our flexible QD-LEDs is below the bending limit of many possible applications, for instance, for smart cards, and further sufficient for large, roll-up, or conformable flat panel displays.[27] This also offers the possibilities of roll-to-roll manufacturing of QD-LEDs and QD-based flexible displays by use of suitable high volume QD-LED processing technologies, such as the emerging QD mist-deposition technique.

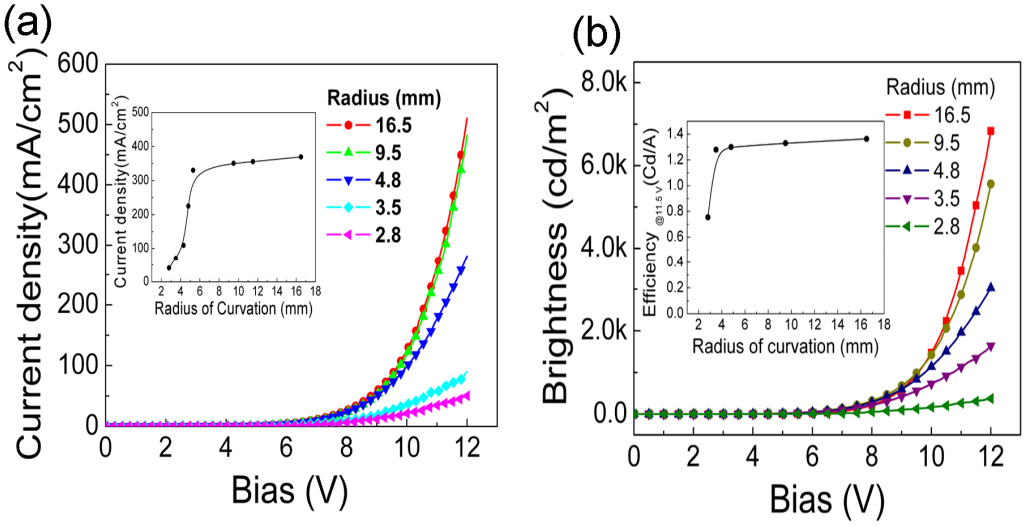

Fig. 12. I-V (a) and L-V (b) curves of red QD-LED worked at different bending state.

In conclusion, colloidal nanocrystal quantum dot base light-emitting diode is a promising new star for next generation displays and solid-state lighting.

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