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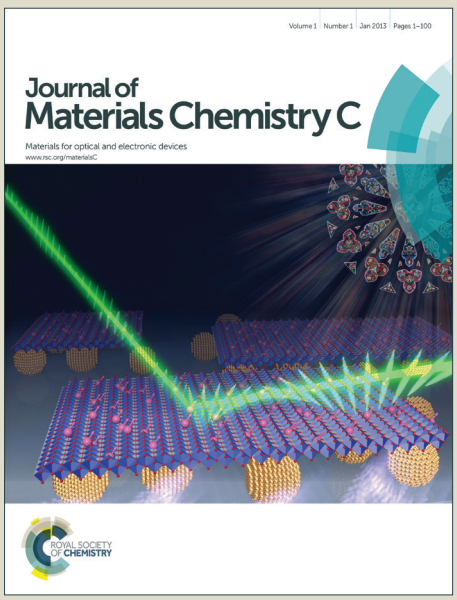

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ARTICLE TYPE

Nanostructured PEDOT:PSS film with Two-dimensional Photonic Quasi Crystals for Efficient White OLED Devices

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A polymeric PEDOT:PSS film nanostructured with Photonic Quasi Crystals that opens the path towards more efficient white OLEDs is presented. For the first time three different quasi crystals families were fabricated (octagonal, dodecagonal and Thue-Morse) onto a conductive polymeric film combining high-resolution electron beam lithography (EBL) and plasma etching techniques to improve light extraction and to control spectral tunability. The efficiency gain obtained in light extraction holds great promise for the use of quasi crystals as functional components in polymeric based White Organic Light Emitting Diode (WOLED) devices.

Introduction

WOLED (White Organic Light Emitting Diode) devices are attracting much attention from researchers all over the world because of their possible use in optoelectronics and in particular in lighting technology.[1-2] The improvements in terms of their external quantum efficiency (EQE) in recent years have been tremendous but still a lot can be done to ensure a key role for this class of devices to save energy. In particular, the use of innovative optical structures such as microlenses, photonic crystals, diffusive films, optical cavities can considerably reduce optical losses due to optical waveguide effects that usually affect multilayered devices.[3-6] All these approaches are usually utilized on devices with a single narrow emission band because of their effect on the spectral emission and the consequence change of the CIE (Commission Internationale de l'Eclairage) diagram.

As it is well known, the organic LEDs have wide band emission and this is even more evident in white-emitting OLEDs in which, there are usually more than one light emitting band. Lately, in order to tune the emission properties of the polymer within the CIE framework, and to improve the device lifetime, it was useful to combine emitting polymers and luminescent dyes i.e. quantum dots,[7] and small molecules.[8] Polyfluorenes and Oxadialzole-carbazole derivates were widely used as polymeric active matrix because of their strong blue emission.[9,10]

On the other hand, photonic quasi-crystals can be employed to help changing the spectra emissions and to improve the efficiency in one or more spectral bands, thus, to contribute to design innovative WOLED architectures. Several tiling geometries have been considered based on different way of structuring the photonic crystal and engineering its band gap properties in view of its use as a practical solid-state light-emitting sources.[11-13] Recent studies[14-16] on photonic quasicrystals (PQCs) with 8-fold (octagonal point group), 10-fold (decagonal), and 12-fold (dodecagonal) rotational symmetries have shown that, in general, higher order rotational symmetry results in an increase of the density of points in the reciprocal space which is important for designing efficient omni-directional outcouplers. Recently, a 12-fold PQC pattern was used to improve the efficiency of light extraction of polymer light-emitting diodes (PLEDs).[17]

In previous works the PC patterns were also directly realized on the anodic electrode to simplify the PC-OLED architecture. In particular, the ITO electrode was partially patterned by focused ion beam (FIB) covered by PEDOT:PSS achieving a good efficiency increase[18] and the low refractive index of PEDOT:PSS[19] gives a high refractive index jump for the ITO-PEDOT:PSS structure that is advantageous also on microstructured[20] or nanostructured grids.[21] Recently, an ITO-free electrode structure has been integrated into a OLED devices utilizing the combination of EBL and PE techniques to fabricate a novel polymeric anode structure based on 2D nanopatterned conductive PEDOT:PSS layers.[22]

In this work, to the best of our knowledge, it is the first time that Photonic Quasi Crystal patterns are realized directly on the dimethylsulfoxide (DMSO) doped PEDOT:PSS polymeric films, and that PQC properties are deeply explored to investigate their potential applications in White OLED devices. Three different PQC families (octagonal, dodecagonal and Thue-Morse patterns)[23-25], are compared to control the spectral emissions and enhance the lighting efficiency of WOLED devices.

Results and Discussion

Photonic Quasi Crystals

Photonic Quasi Crystals[26-30] are a class of structures that do not have translational symmetry, but obey local rotational symmetry, very often with symmetry axes forbidden for regular crystals, e.g., five-fold rotational symmetry. Originally discovered for rapidly annealed metallic alloys by Shechtman,[26] photonic quasi crystals are man-made structures. As quasi crystals combine both, long-range order as in photonic crystals (although not in a repeating fashion) and local arrangements of atoms in fixed positions but with different configuration of the surrounding atoms like in glasses, they promise to possess unusual optical transport properties. The structural complexity of PQCs is measured by their spatial Fourier spectra, which are discrete (singular) for quasiperiodic systems, singular-continuous, or absolutely continuous for pseudorandom structures of increasing complexity.

The Thue–Morse (TM) sequence is an example of an aperiodic structure with a singular continuous contribution to the diffraction pattern. To construct the 2-D TM structure, a one-dimensional binary TM sequence is firstly constructed using the following rule. First of all, let's give an arbitrary sequence of two symbols, A=0 and B=1, and then a new sequence is formed by replacing each occurrence of A with the pair (A, B) and each occurrence of B with the pair (B, A).[31] This sequence is neither periodic nor quasiperiodic. By inflation rule the one-dimensional aperiodic lattice can be generalized to the two-dimensional space introducing a substitutional matrix obtained as Cartesian product of 1D TM sequences.[25, 32-33]

In this work, the 2D TM structures have been experimentally obtained by removing particular lattice points from a regular square array with lattice constant a by EBL as described in the experimental section. The selected units are chosen in agreement with the inflation rules encoded into the substitutional matrix of the 2D TM sequence. The centres of the air filled rods were located at the vertices of a 2D TM lattice of order N=10.

The fabricated octagonal structure was determined by simulating the quasi periodic transverse irradiance distribution given by 8-beam interference process and was theoretically analyzed through finite difference time domain (FDTD) simulations of the transmittance spectra.[34]

The 12-fold quasicrystal structure proposed by Zoorob in 2000 grown by use of the random Stampfly inflation method, is based on a dodecagon composed of an inner hexagon consisting of six equilateral triangles surrounded by a ring of squares and equilateral triangles. Our structure is a "triangle–triangle" crystal configuration with a rod radius to pitch length ratio (r/a) of 0.25.

Nanopatterned PEDOT:PSS Film Fabrication

Our method to directly pattern the highly conductive[22] PEDOT:PSS films together with the whole fabrication process is described in figure 1. PEDOT:PSS-quasi periodic nanostructured arrays were fabricated by electron–beam lithography (EBL) on glass substrates. The realized two-dimensional photonic quasi crystals are made of air rods embedded into the organic matrix of styrene methyl acrylate based polymer (ZEP) with lattice parameters calculated with numerical simulations.

Such nanostructured ZEP films were used as a lithographic mask during the anisotropic plasma etching process. Through this process, six PQC nanostructures were realized using the

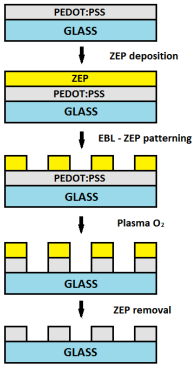

Fig.1 Schematic diagram-process for the highly conductive PEDOT:PSS-PC fabrication.

following arrangements: A) Octagonal, B) Dodecagonal, C) Thue Morse of order M=10.

In particular the fabricated structures are: octagonal patterns with air rods of diameters d=250 nm, d=500 nm and d=750 nm, a dodecagonal pattern with air rods of diameter d=500 nm and lattice constant a=1000 nm, and Thue Morse patterns of order M=10 with air rods of diameters d=500 nm and d=750 nm and lattice constant a=720 nm and a=1080 nm respectively.

White Polymer Fabrication

In order to establish the contribution of the PQC structures on White OLED devices we prepared organic white-light-emitting blend materials and then we spinned them on the nanostructured anodes. These polymers were selected also because they were already used in polymer light emitting diodes.[35] Initially, thin films (~70nm) with different concentration of Poly[2-methoxy-5-(3′,7′-dimethyloctyloxy)-1,4-phenylenevinylene] (MDMO-PPV) in polyfluorene oxadiazole (PFO) polymer were prepared by spin-coating on glass substrates in order to optimize the concentration of MDMO-PPV to obtain white emission. It was observed that the optimal concentration to obtain white emission is with 2 wt% of MDMO-PPV in PFO polymer. Figure 2 shows photoluminescence intensity of films containing PFO and PFO with 2 wt% of MDMO-PPV.

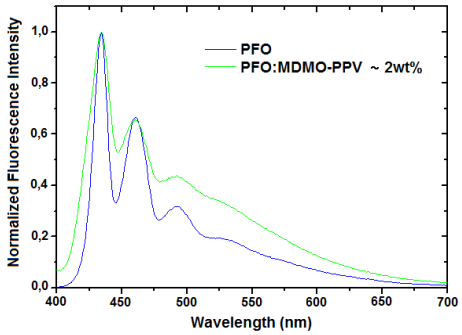

Fig.2 Fluorescence spectra of pure PFO (blue curve), and PFO:MDMO-PPV (green curve) films with an excitation at 380 nm.

Morphological and Optical Characterization

The PQCs polymeric nanostructures realized were characterized in the direct space through metrological measurements with scanning electron microscopy (SEM) (figure 3).

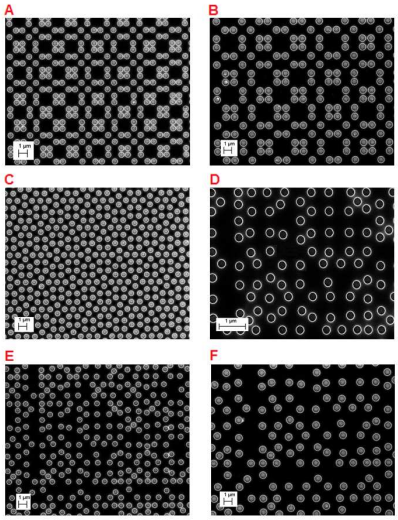

Fig.3 SEM images of the PQCs obtained after the EBL ZEP exposure on the PEDOT:PSS highly conductive layer: A) Thue Morse d=500 nm, B) Thue Morse d=750 nm, C) Dodecagonal, D) Octagonal d=250 nm, E) Octagonal d=500 nm, F) Octagonal d=750 nm.

An optical setup was adopted to evaluate the PQCs properties at room temperature and to have an estimation about the extracted spectrum[36,37] of the different lattices which better matches the desired properties of the final OLED. The set-up to measure the light propagating in the glass substrate which is extracted by diffraction is reported in figure 4. The PQCs-based polymeric structures were tested introducing a white light from the edge of the glass substrate that propagated inside the glass due to the total internal reflection phenomena.

Study of Light Extraction

The vertical extracted spectra (0 degree) of the nanostructured films are shown in figure 5a. All the spectra have an extracted background into the whole visible region and different extraction bands can be distinguished in the analyzed signal for all the samples realized. The full width half maximum was mainly between 30 and 50 nm for each band observed. Figure 5b shows the radial properties of the emission pattern for the different PQCs. Lattice symmetry not only has an important effect on extraction efficiency and beam directionality but also on the quality of the far field emission pattern.

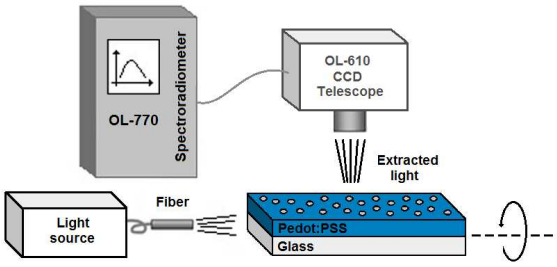

Fig.4 Set up employed for the spectral evaluation of the PQC-nano arrays: our sample is illuminated from an edge of the glass with a white light. The light propagating in the glass substrate which is extracted by diffraction is measured at room temperature using a multimode fiber, a CCD imaging telescope (OL610), and a CCD-based spectroradiometer (OL770-LED).

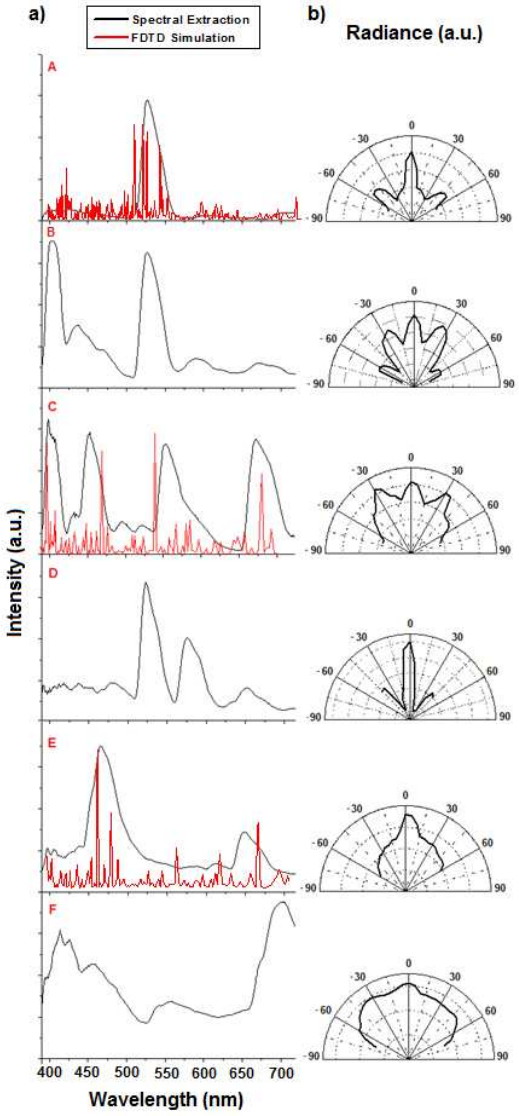

Fig.5 Spectral extraction (a), angular radiance (b) and FDTD simulations (red lines) of the structures: A) Thue Morse d=500 nm, B) Thue Morse d=750 nm, C) Dodecagonal, D) Octagonal d=250 nm, E) Octagonal d=500 nm, F) Octagonal d=750 nm.

In order to achieve the enhancement of out-coupling efficiency through an appropriate chip design, we performed the calculation of the resonant frequencies for a single pattern belonging to each family of the quasi-crystals considered by 3D numerical simulations (red line in figure 5a). The results obtained have provided indications concerning the possibility to get extracted peaks in the visible region. We employed a finite difference time-domain numerical method (FDTD) performed with a commercial software (Full Wave simulation tool, RSoft Design Group, Ossining, NY). We have assumed that the glass substrate (corning eagle 2000) and air are semi-infinite with a constant refractive index n_{glass}=1.51 and n_{air}=1, whereas the dispersion relation in the spectral region of interest has been used for the PEDOT:PSS film. The thickness of the polymer and depth of the holes used is $t_{pedot:pss}$= t_{holes}=160 nm. During the calculations, we used a time step (in units of ct) of 10-2μm and a spatial grid with step size of 20 nm for each direction. Periodic boundary

conditions on the x−z plane of the nanostructure and perfectly matched layer boundary condition (PML) on y direction were used for the dodecagonal and Thue-Morse structures while PML conditions were used in all directions for the octagonal structure, since it is difficult to design a periodic cell on this kind of pattern. In particular, we used a rhombic unit supercell for the dodecagonal pattern and an M4 order supercell for the Thue-Morse pattern.[31] The simulated structures were excited from the air by a short impulse with a broad spectrum and plane wavefront. It can be noticed that the experimental resonances exhibit a good match with the simulated peaks.

Starting from the simulated data, we scaled octagonal and Thue-Morse pattern in order to have more and different extracted bands in the region of interest. In particular, for the Thue Morse structure we increased the size of the holes trying to obtain more peaks in the visible region, while, referring to the octagonal structure, we have both increased and decreased the holes size to evaluate the displacement of the two peaks highlighted by the first structure realized.

The dodecagonal pattern shows many extracted pathways with the selected lattice parameters. Figure 5b shows how the geometrical parameters affect both the extracted spectra and the angular radiance profiles. In particular, the background of the extracted light increase as the sizes of the pattern increase and make the structure more diffusive with a more isotropic angular profile extraction. Moreover, the CIE diagram (figure 6a), associated to A, C and E spectrum in figure 5a, shows that the dodecagonal photonic structure has an extracted spectrum well localized in the white region, close to x=0.33 and y=0.33 coordinates, while the octagonal and the Thue-Morse nanostructures could be further optimized.

Figure 6a shows the spectrally integrated light intensity characteristics related to all the realized structures and give us the opportunity to select the appropriate structures to be used to enhance efficiency in a white OLED device. In this regard, it is possible to observe that the structures B (Thue Morse d=500 nm, a=1080 nm), C (dodecagonal) and F (octagonal, d=750 nm) are closer to the white point.

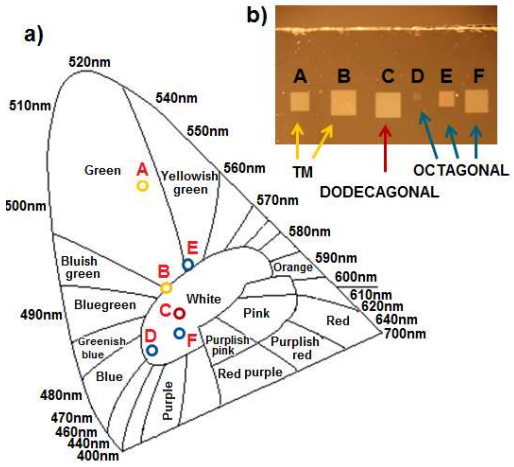

Fig.6 a) CIE diagram of the light extracted from the PQC anodes: Octagonal -blue points, Dodecagonal - red point and Thue-Morse - yellow points, b) Optical microscope image of the light extracted from the PQC structures.

The image in figure 6b obtained with an optical microscope shows the extracted light by PQCs areas while white light propagates into the glass substrate. The PQCs areas were seen almost white for all the fabricated structures because of the wavelength-selective diffraction by the quasi-periodic nanopatterns. In the case of white sources, to achieve a large enhancement of light extraction, it is important to explore all the physical parameters that modify the light propagation via diffraction on nanometric patterns and relate them to the emission bands of the white source. The experimental results demonstrate that the realized photonic structures are then perfectly transferred onto the PEDOT:PSS films and that our method may offer the possibility to exploit a large variety of new functionalities of the innovative nanostructures.

We repeated the experiment on the structures B, C and F after covering them with the organic white-light-emitting blend material (PFO/MDMO-PPV) by spin coating process.

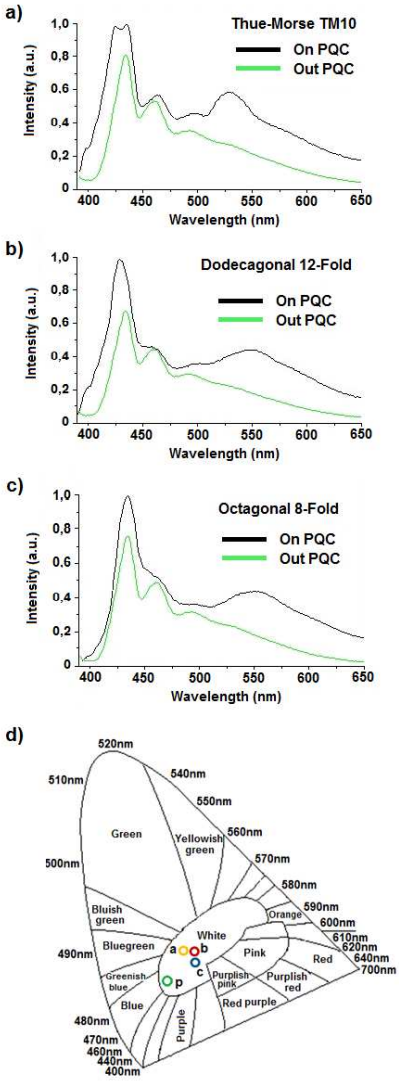

Fig.7 Spectrum measured with PFO/MDMO-PPV and extracted from the PQCs (straight lines) and out of the PQCs (dot lines): a) Thue Morse, b) Dodecagonal, c) Octagonal, d) CIE diagram relative to the spectrum a,b,c, and of the PFO/MDMO-PPV polymer (p).

We used a UV led as light probe (wavelength peak 360 nm) to stimulate the fluorescence of the polymer. We analyzed the fluorescent spectra coming from the nanostructured areas (straight lines, figure 7) and from the nearby non-structured areas (dot lines, figure 7). In this way we can predict the enhancement resulting from the use of PQC structures in combination with an emitting polymer that might be used in WOLED stacked devices. In particular, we observed an enhancement in the light extracted of 55%, 66% and 68% related to the octagonal, Thue-Morse and dodecagonal structures, respectively, calculated by the formula $[(R_{outPQC}-R_{inPQC})/R_{inPQC}]*100$, where R is the measured spectral radiance.

It is worthwhile to notice that with the use of the proposed architectures for a WOLED device it is also very important to achieve holes in the designed patterns of depth that does not affect the electrical continuity, so avoiding reaching the glass.[38] Moreover, the radiation emitted by the device is further affected by the presence of the cathode.[39]

In summary, we demonstrated the possibility to realize a PQC directly on the polymeric film surface combining EBL and a Plasma Etching (PE) process to partially structure the PEDOT:PSS. We performed several measurements and simulations to elude the trial and error approach and to investigate the quasi-photonic structures according to the fluorescent spectra of the emitter polymer, to easier achieve a real enhancement of light extraction. Thus this kind of characterization allows us to compare and evaluate the enhancement of the extracted light in different quasi periodic/deterministic aperiodic patterns in white OLEDs application without necessarily produce a great number of test devices.

Experimental section

Nanopatterned organic film fabrication: Our method to directly pattern the organic films together with the whole fabrication process is described in figure 1. PEDOT:PSS-quasi periodic nanostructured arrays were fabricated by electron–beam lithography (EBL) on glass substrates. Highly conductive PEDOT:PSS (160 nm) was spin coated onto a corning glass substrate. Such highly conductive film was obtained by doping the PEDOT:PSS with DMSO as described in a previous paper.[40] Then, a 200 nm thick layer of a styrene methyl acrylate based polymer (ZEP), which is electron-sensitive, was spin–coated on top of the cleaned PEDOT-PSS surfaces. Thereafter, the resist films were dried in oven at 170°C for 5 min. An e–beam direct writing system (Raith150) was used to define the pattern operating at 10 keV with electron dosage of 24 pA. Finally, nanostructured arrays were formed after developing in n-Amyl acetate for two minutes. Such nanostructured ZEP films were used as a lithographic mask during the anisotropic plasma etching process performed by high-vacuum Plasma Enhanced Chemical Vapor Deposition (PECVD) system (Plasmalab800+, Oxford Inst.) operating at 13.56 MHz with these parameters: power density around 270 mW/cm^2, 200 sccm for the O_2, and pressure 400 mTorr for 10 minutes of treatment at 100 °C. As final step, the anisole solvent was used to remove the ZEP without damaging the underlying PEDOT:PSS film.

Functionalization of nanostructures with white polymer: Organic white-light-emitting blend materials were prepared by mixing PFO and MDMO-PPV as the blue host and the red guest emitting materials, respectively. PFO was dissolved in chlorobenzene with a material content of 15mg/ml and MDMO-PPV was added at various concentrations (1, 2 and 5 wt%) in the same solvent.

PFO films with 2 wt% of MDMO-PPV were deposited by spin coating on the nanopatterned DMSO-PEDOT:PSS substrates. These films were then annealed at 120°C in oven for 2 hours.

Measurement set-up: The nanostructured substrates have been characterized by Scanning Electron Microscopy and out-of-plane diffraction measurements. The set-up to measure the light propagating in the glass substrate which is extracted by diffraction (figure 4) is composed by a multimode fiber, a CCD imaging telescope (OL610), and a CCD-based spectroradiometer (OL770-LED). The sample is placed on a rotating holder which allows the angular resolved measurements. The PQCs-based polymeric structures were tested introducing a white light from the edge of the glass substrate that propagated inside the glass due to the total internal reflection phenomena.

Conclusions

In conclusion, we have demonstrated experimentally and numerically PQCs polymeric film properties. As a result, spectral modifications and emission enhancement of polymeric WOLED based devices can be optimized with the presented study. We demonstrated that the PQCs can be designed to match the properties of the active photonic material by changing both arrangement and the geometrical parameters of the structures to obtain, eventually, enhanced performances of the PQC based device. We simulated and then selected three different arrangements of photonic quasi-crystals exhibiting a good enhancement in light extraction in the wavelength range overlapping the photoluminescence emission of the fluorescent polymeric layer. Finally, it has been shown that using PQCs nanostructures in a highly conductive PEDOT:PSS polymeric film a remarkable efficiency gain is obtained with an improvement higher than 60% for white light emission.

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‡ Footnotes should appear here. These might include comments relevant to but not central to the matter under discussion, limited experimental and spectral data, and crystallographic data.

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Table of Contents entry

Polymeric nanostructured highly conductive films by means of Photonic Quasi Crystals (PQCs) topologies are presented envying the possibilities to open the path towards high efficient white OLEDs. They exhibit a good enhancement in light extraction related to the fluorescent white polymeric layer reaching an improvement higher than 60%.