

Advanced Engineering Materials Progress Report on

Fluorescence and Phosphorescence in Organic Materials**

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Progress reports are a new type of article in Advanced Engineering Materials, dealing with the hottest current topics, and providing readers with a critically selected overview of important progress in these fields. It is not intended that the articles be comprehensive, but rather insightful, selective, critical, opinionated, and even visionary. We have approached scientists we believe are at the very forefront of these fields to contribute the articles, which will appear on an annual basis. The article below describes the latest advances in fluorescence and phosphorescence in organic materials.

1. Introduction

Fluorescent and phosphorescent materials continue to attract commercial and scientific interest since not only can they be used to fabricate light-emitting diodes (LEDs) and large area flexible displays, but they are now approaching efficiencies that may even allow for applications in panel lighting. In recent years, our ability to understand, control and tailor luminescent conjugated materials has improved dramatically. The highly efficient organic LEDs that can now be produced^[1-3] are becoming an attractive alternative to those based on conventional inorganic luminescent materials.^[4]

However, in order to advance the emission properties of this relatively new class of materials, a few fundamental issues must still be addressed and eventually resolved. These issues include the mechanisms of formation, transport, and harvesting of spin singlet and triplet excited states, as well as how these mechanisms are influenced by the morphology and structure within films of luminescent materials. Through understanding and controlling the microscopic order, which establishes how excited states interact within and between molecular sites, it is possible to modify and improve the macroscopic emission characteristics such as color and efficiency.

2. Fluorescence and Phosphorescence under Electrical Excitation

2.1. Controlling Efficiencies and Color with Dopants

While there have been some improvements in LED efficiencies through modifying the overall device architecture

such as better hole-injecting layers for more balanced charge injection, much of the research in the last year has focussed on using both types of excited states towards light emission, that is spin singlet and spin triplet states. Statistically, 75 % of injected charges are in the three-fold degenerate triplet state, while only 25 % are in the singlet excited state.^[4] In contrast to singlet states, the triplet states of hydrocarbon materials do not usually emit light since hydrocarbon materials have no mechanism to promote the spin-flip needed for an optical transition.^[5] Consequently, much effort has been devoted to using materials that have significant spin-orbit coupling, the prime example being organometallic complexes.^[2,3,6-13] These complexes are doped into wide energy gap organic host materials where balanced charge injection and efficient transport and recombination can occur. The majority of the host's non-emissive triplet excitons can then be harvested either by Förster transfer to the singlet state of the guest complex followed by intersystem crossing to the guest triplet state or directly by Dexter transfer from the triplet of the host to that of the guest giving high device efficiencies as demonstrated by Baldo et al.^[1] Since these first reports for the phosphorescent complex 2,3,7,8,12,13,17,18-octaethyl-21H,23H-por-

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[**] Also published in *Advanced Materials: Adv. Mater.* **2002**, *14*, 701.

phine platinum(II) (PtOEP), the importance of using triplet confinement layers and the possibility of direct formation of triplets on the guest have become clear.^[3] Triplet excited states have long lifetimes, typically in the microsecond to millisecond range, so that triplet-triplet annihilation and concentration quenching on the guest complex can reduce the LED efficiency, especially at high current densities.^[8,9,12,13] This effect can be reduced when spacer groups are included on the complex that introduce steric hindering and so limit the interaction of the lumophores.^[13] Alternatively, the triplet lifetime of the complex can be changed by choosing a different metal center,^[8,12] or schemes involving energy transfer to a second fluorescent dopant with a short lifetime can be used.^[9]

Iridium-based complexes are a popular choice for the triplet harvesting dopant as these compounds have a short triplet life-

time of only a few microseconds compared to at least 50 μs for PtOEP.^[12,13] Recent reports from Adachi et al.^[3] have demonstrated that energy harvesting using iridium-based complexes can be so efficient that temperature-independent internal phosphorescence efficiencies of 87% are achieved. Consequently, the external efficiency of the LED is no longer limited by the emission efficiency of the organic material itself, but instead by the out-coupling efficiency of their device structure, which is 22%.^[3] These results are shown in Figure 1 where the efficiency of the devices as a function of current and the calculated out-coupling efficiency are given. The maximum external quantum efficiency is a very impressive $19 \pm 1\%$ (60 ± 5 lm/W). This is higher than the efficiency of tungsten lamps (typically less than 20 lm/W) and approaches that of fluorescent tubes (typically 70 lm/W). For these systems it is possible to tune the emission color simply by changing the ligands on



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Joanne Wilson was born in Leamington Spa, England, in 1976. She graduated in physics from Cambridge University in 1998, and has recently completed her Ph.D. in the group of Richard Friend. Her Ph.D. work focussed on using platinum-containing conjugated polymers as a model system for studying the photophysics of triplet states.



Richard Friend has been on the Faculty in the Department of Physics, University of Cambridge since 1980 where he is the Cavendish Professor of Physics. Professor Friend has developed the study of organic polymers as semiconductors, and has demonstrated that these materials can be used in a wide range of semiconductor devices, including light-emitting diodes, transistors, and photocells. He has been active in the process of technology transfer of this research to the development for products. He co-founded Cambridge Display Technology Ltd. in 1994, which is developing polymer LED displays, and co-founded Plastic Logic Ltd. in 2000, which develops directly printed polymer transistor circuits. He has published more than 600 papers on organic semiconductors and related research areas, and is a named inventor on more than 20 patents and patent applications.

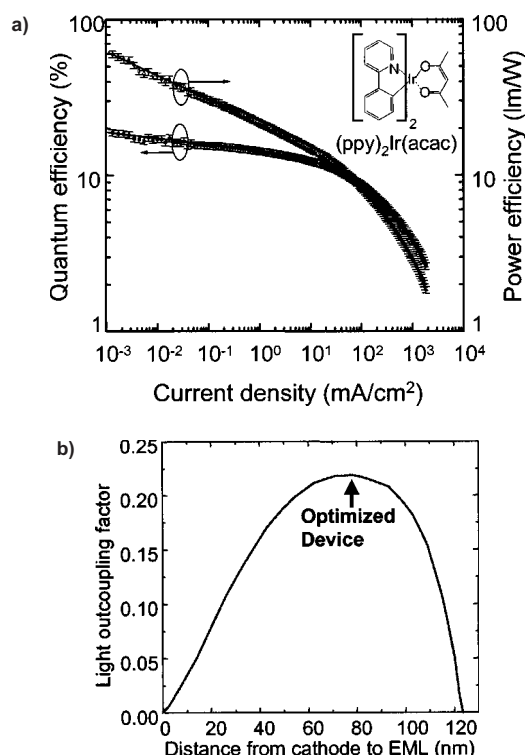


Fig. 1. The device characteristics of an organic LED doped with the complex $(ppy)_2Ir(acac)$. a) The external quantum and power efficiencies. A maximum external quantum efficiency of $19 \pm 1\%$ and power efficiency of 60 ± 5 lm/W were obtained. The inset shows the molecular structure of $(ppy)_2Ir(acac)$. b) The calculated light out-coupling factor for the device structure used as a function of the distance of the light emitting layer from the cathode. Reproduced with permission from Adachi et al. [3]. Copyright 2001 American Institute of Physics.

the complex, and many examples of different color emission (from blue to red) have been reported,^[3,8-10,12] suggesting that this may be an efficient way to achieve white-light emission for lighting applications in the future.

While heavy metal containing complexes can be used to harvest non-emissive triplets and optimize emission efficiencies, a different choice of metal complexes makes it possible to shift the color of emission to the near or far infrared, a wavelength range that is otherwise difficult to access with organic lumophores. These rare-earth complexes, which emit in the infrared, are increasingly being incorporated into organic host matrixes.^[14-19] In contrast to the metal complexes discussed above, the optical emission from rare-earth complexes is not due to transitions based on the organic ligand, but instead arises from transitions within the f-shell on the rare-earth ion itself. This results in very sharp red, or infrared, emission bands.^[16] The narrow emission from an Eu complex is shown in Figure 2. To give some more examples, the emission of Nd^{3+} is at $1.34 \mu m$ and that of Er^{3+} is at $1.53 \mu m$.^[18] These wavelengths are compatible with existing optical fiber telecommunications technology,^[18] and rare-earth complexes are also of interest for use as phosphor screens for field-emission displays.^[17] Another tempting possibility is that the energy level structures of rare-earth ions appear to be suitable for producing electrically pumped lasers.^[15]

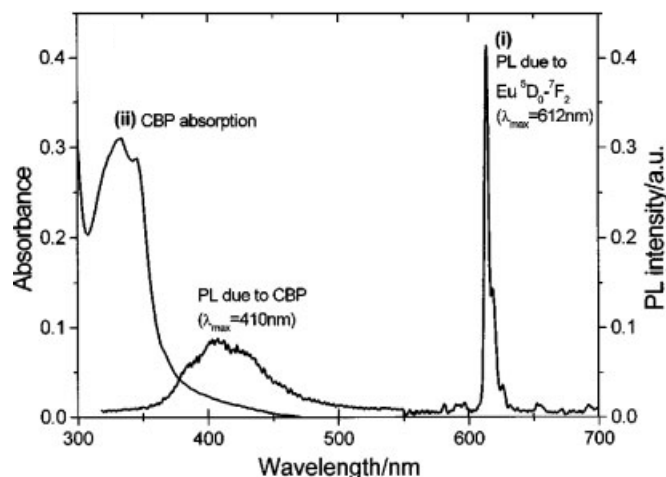


Fig. 2. The emission spectra from the rare-earth complex $Eu(TTA)_3phen$ (TTA =the-noyltrifluoroacetone, $phen$ =1,10-phenanthroline) doped in a CBP host (CBP=4,4'-N,N'-dicarbazole-biphenyl). The absorption of the host is also shown. The narrow emission band (at 612 nm) is characteristic of rare-earth complexes. Reproduced with permission from Adachi et al. [19]. Copyright 2000 American Institute of Physics.

2.2. Understanding Phosphorescence

The harvesting of triplet excitons has proven to be a very successful way of making efficient organic LEDs. To make this an effective process, there are two major requirements. It is necessary to know where the triplet energy level is in the organic compound, and it is important that there are enough non-emissive triplet excitons produced that they do actually limit the efficiency of the device in the first place. A better understanding of what controls these properties will enable the fabrication of more efficient LEDs, and on both fronts there have been significant advances in the last year.

A major breakthrough a few years ago was the discovery that, for electrical excitation of conjugated polymers, higher numbers of singlet excitons are generated than would be expected from the 25% statistical limit.^[20,21] The first measurements gave singlet generation fractions of 50% and now it would appear that for some materials the fraction can be significantly higher than this. So, last year, the fundamental question of what actually determines the fraction of singlet and triplet states that are generated was addressed. Several suggestions have been put forward, both from experimentalists and theoreticians.^[22-27] One model is that the formation rates for singlet and triplet excitons are affected by intermolecular charge transfer and recombination processes.^[23,27] These processes have contributions from both the interchain one electron transfer matrix elements (the probability of jumping from one chain to the next) and the two electron bond charge integrals (the electron repulsion between electron density around a bond on one chain and a site on an adjacent chain). Wohlgenannt et al.^[22] have also suggested a dependence of the singlet generation fraction on the covalent or ionic nature of the bonds, and therefore the optical gap. Their experimentally determined ratios of singlet to triplet formation cross sections for a number of conjugated polymers

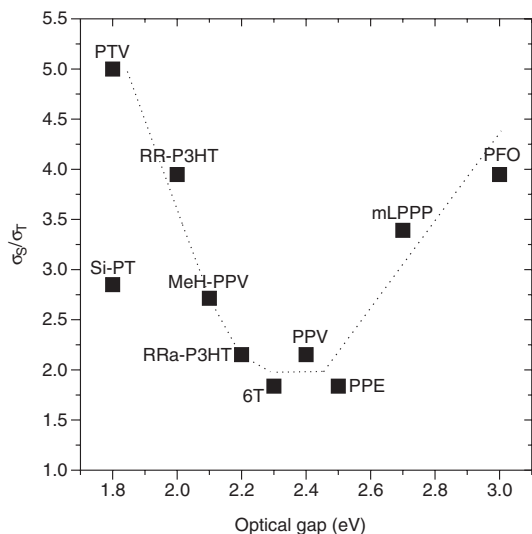


Fig. 3. Experimentally determined ratios of singlet to triplet formation cross sections for several conjugated polymers as a function of optical gap. The dotted line is a guide to the eye. Singlet generation fractions may be obtained from these ratios, since the singlet generation fraction is equal to $(1+3/(\sigma_S/\sigma_T))^{-1}$. Reproduced with permission from Wohlgenannt et al. [22]. Copyright 2001 MacMillan Publishers Ltd.

are plotted as a function of optical gap in Figure 3. Another view is that the relative energies of the original polaron states and the singlet and triplet states to be formed are important,^[24] and that the dissipation of energy into the lattice on formation of singlet and triplet states would favor the formation of singlets.^[27] Since the singlet state is generally more delocalized than the triplet, the better overlap it has with the initial charge separated excited state may also affect the singlet generation fraction.^[27] Other work compared the singlet generation fraction in a conjugated polymer and its corresponding monomer and found a significant difference in the singlet generation fraction for the two types of materials.^[28] A different, spin-dependent mechanism appears to be at work in polymers, which favors singlet formation so that the 25% limit does not apply, while for small molecules the singlet generation fraction remains limited to 25%.

So, while for small molecule LEDs, the future is indeed to efficiently harvest energy from non-emissive triplet states, for polymer LEDs high efficiency emission may be achieved purely from singlet states. In fact, for polymers, the important question is how the singlet formation mechanism can be controlled in order to tune the generation fraction as required. Undoubtedly, research in the coming years will shed more light on the details of the generation mechanism of singlet and triplet states in organic materials.

If one wishes to use triplet harvesting techniques, it is extremely useful to know the energy of the non-emissive triplet states in the host materials. Thanks to the development of techniques such as time-resolved detection,^[29–31] optically detected magnetic resonance,^[32,33] electron paramagnetic resonance,^[34,35] pulse radiolysis, and energy transfer measurements,^[36,37] and the substitution of heavy metal atoms into the conjugated polymer backbone^[38,39] there has been a lot of work on triplet states that were previously inaccessible. As a

result, much more is now known about the energy levels and dynamics of triplets. It has become clear that the singlet S_1 and triplet T_1 energy levels in conjugated polymers are separated by a finite exchange energy which is around 0.7 eV.^[29,36–39] (This energy increases for oligomers as the singlet exciton is confined.) It is therefore possible to estimate the energy of the non-emissive triplet states purely from knowledge of where the emissive singlet state lies.

The processes of intersystem crossing and internal conversion have also been investigated and a fuller understanding of the mechanisms and rates involved are now emerging.^[25,40–42] For optical and electrical excitation, the intersystem crossing process between singlet and triplet manifolds can be a significant non-radiative decay mechanism and limits the overall fluorescence efficiency. The rate of intersystem crossing is controlled by the relative energies of the lowest excited singlet state S_1 and the closest energy triplet state T_n , and changes with oligomer length since the energies of S_1 and T_n shift.^[40] (It is enhanced by heavy atom induced spin-orbit coupling or suitable vibrational coupling such as ring twists.^[40]) Similarly, the internal conversion rate from the lowest triplet excited state T_1 to the singlet ground state S_0 increases exponentially with decreasing energy separation between the two states.^[42]

3. Interactions of Excitations within Luminescent Materials

3.1. Understanding Exciton Dynamics

For electrical excitation, the exciton formation mechanism in conjugated polymers may be linked to interchain interactions.^[23,27] In fact, one of the other remaining important questions is exactly how electrical charge and excitation energy are transported along and between chains and on what time-scale this occurs.^[43,44] Charge and energy transfer affect a variety of processes such as triplet-triplet annihilation,^[30,31,45] delayed fluorescence,^[46] and exciton dissociation,^[47,48] relaxation, and transport.^[49]

In addition, excitation energy can transfer to aggregate or excimer sites where an unsuitable relative orientation of adjacent interacting polymer chains may lead to only weak radiative coupling between the lowest excited state and the ground state, and consequently low emission efficiencies.^[27,43] Such unfavorable π -interactions can be reduced if the wavefunctions involved are very delocalized, the chains are well separated or if the chain axes are aligned in a perpendicular or staggered fashion.

Past approaches to combat excimer and aggregate formation have focussed on adding bulky side chains to polymer backbones,^[43] and now light-emitting dendrimers are being used as a model system to investigate the effect of introducing a controlled amount of insulation between the lumophores.^[50] The reduction in charge transport with increasing dendrimer generation is compensated for by an increase in

photoluminescence efficiency, such that increasing the amount of insulation increases the efficiency of LEDs made from these materials. Recent studies have addressed the control of energy transfer along polymer chains by ordering and isolating them from each other by putting them into a non-emissive porous silica host matrix as shown schematically in Figure 4.^[44] The matrix was designed with 22 Å pore diameter so that only one poly(2-methoxy-5-(2'-ethyl-hexyloxy)-1,4-phenylene-vinylene) (MEH-PPV) polymer chain could fit into each channel and the emission was seen to be polarized along the pore direction. It was clear from this work that intrachain energy transport is much slower than interchain transport as the dipoles are head to tail along the chain so Förster transfer is not efficient. These experiments have elegantly demonstrated that the transport and trapping of excitation energy can be controlled with different molecular architectures.

Thanks to the development of new techniques for controlling the conformation of polymer chains it has now also been possible to study what effect different conformations have on charge and energy transport, and on the emission characteristics.^[51–53] By attaching surfactant molecules to a poly(*p*-phenylene-ethynylene) chain, Kim et al.^[51] were able to mechanically induce reversible conformational changes and to relate the optical properties of the polymer to interchain interactions and the single chain conformation. Muccini and co-workers demonstrated that the mechanism of energy transfer in α -sexithienyl is highly sensitive to the molecular environment.^[53] Through comparing sub-mono-layer and lamellae architectures they were able to relate features seen in the photoluminescence spectra to precise structural topologies.

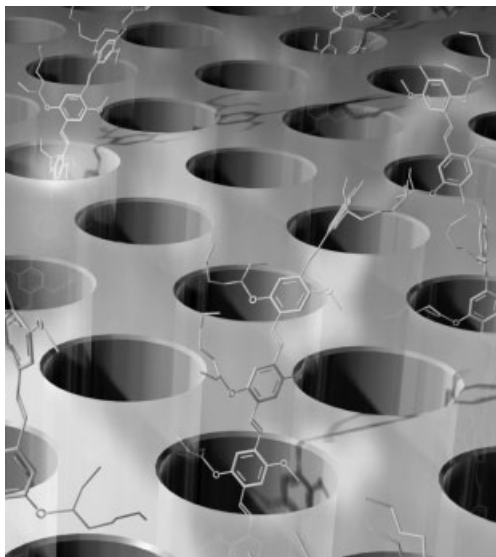


Fig. 4. Schematic representation of single polymer chains embedded in the channels of an ordered mesoporous silica glass as in the experiment of Nguyen et al. [44]. The poly(2-methoxy-5-(2'-ethyl-hexyloxy)-1,4-phenylene-vinylene) (MEH-PPV) chains within the channels are aligned and isolated from each other; the chain tails that extend out of the pores are randomly orientated and can be in contact with each other. Reproduced from Nguyen et al. [44].

3.2. Controlling Excitations with Structural Order

Since microscopic properties determined by the morphology of organic films are systematically translated into macroscopic emission characteristics, an effective approach for controlling these emission characteristics is to use aligned or self-assembled structures. Methods for achieving ordered films of conjugated polymers have improved such that polarized emission for display applications can now be achieved with electrical excitation.^[54] Various techniques have been used to obtain these ordered films, such as mechanical alignment by stretching or rubbing, Langmuir–Blodgett deposition, liquid crystalline self-organization, and the use of pre-aligned substrates.^[54–58] An interesting LED architecture that was reported recently involved a lower layer of aligned, green-emitting poly(*p*-phenylene vinylene) (PPV) and an upper layer of perpendicularly aligned, red-emitting poly(3-(6-methoxyhexyl) thiophene) (P6Me).^[59] The emission was seen to be either green or red depending on the orientation of a polarizer placed above the device, demonstrating that it is possible to obtain polarized light with an easily variable color over a wide spectral range. Polarized light has also been obtained from epitaxially grown crystals of thiophene/*p*-phenylene co-oligomers,^[56] and circularly polarized emission has been reported for polymer LED structures that include a chiral nematic liquid crystal reflector.^[60]

The fact that it is now feasible to achieve alignment even for structures in which electrical excitation occurs opens up new avenues for polymer lasers. Although solid-state optically pumped lasing has been demonstrated for many different conjugated polymers,^[61] an electrically pumped laser based on these materials has yet to be made. In order to make electrically pumped lasers possible it is necessary to find ways to reduce the lasing threshold and to increase the efficiency of emission, for example by moving the energy of emission away from that of absorption.^[62] Many of the most recent reports of optically pumped lasing make use of alignment, and often of liquid crystalline properties.^[61–63] Yet the soft, flexible nature of conjugated polymers also makes it possible to pattern devices with wavelength-scale structures using soft lithography and embossing techniques. These structures can modify the generation and propagation of light within the materials and allow for new architectures, such as polymers in the form of circular gratings that act as surface-emitting distributed Bragg reflectors and lase under optical excitation.^[64]

In fact, the patterning of conjugated polymer films also opens up avenues to improve the out-coupling of light from light-emitting diodes. An example of this is the corrugated LED structure fabricated by Matterson et al. that scatters waveguided light out from the device before it can be reabsorbed.^[65] This structure results in an increase of the electroluminescence efficiency by a factor of two and also has the advantage of producing polarized emission. Another approach to improving light output is the use of a silica aerogel layer with a low refractive index (1.01–1.10) between the

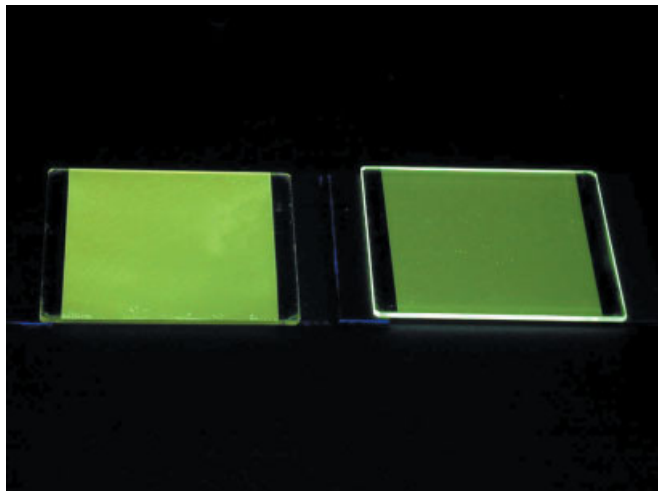


Fig. 5. A photograph comparing ultrathin emissive organic layers on glass substrates with (left) and without (right) an aerogel spacer layer under UV irradiation. Poor out-coupling and waveguiding due to internal reflections causes the intense edge emission seen in the right hand sample. This does not occur when the aerogel spacer layer is added (left hand sample). Reproduced from Tsutsui et al. [66].

polymer layer and the glass substrate in order to modify the angle for total internal reflection at the interface.^[66] Figure 5 compares emissive organic layers on glass with and without a silica aerogel layer, and the amount of waveguiding that occurs has clearly been reduced by the insertion of this low refractive index material. This out-coupling technique has been shown to double the amount of light that can be emitted from polymer LEDs with the advantage that the underlying device structure does not need to be modified. So with the high efficiencies of 60 lm/W that have been reported last year^[3] it is clear that it may already be possible to achieve 120 lm/W from organic LEDs by using this out-coupling technique.^[66]

4. Outlook

The advancements made in the understanding and control of organic fluorescent and phosphorescent materials during the past year are sure to lead to improved light-emitting structures including more efficient LEDs and electrically driven lasers. Work last year has demonstrated that the efficiencies of organic based devices can already be competitive with those of their inorganic counterparts. Critical for this development will be the further growth in the understanding of the electronic properties of organic semiconductors and the control of exciton dynamics. Recent research has demonstrated the importance of spin-interactions for the formation of excited states and, in the near future, we expect that detailed models will improve our understanding of the quantum mechanical processes of the formation of emissive states and allow for tailoring of branching ratios between fluorescent and phosphorescent states. In the longer term, research is moving from optimizing the efficiency of emission within the organic layer of the LEDs to optimizing the out-coupling of

light from the device structure by incorporating microcavities, shaped substrates, or an index matching medium.

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