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Morphology, structure and photophysics of thin films of perylene-3,4,9,10-tetracarboxylic dianhydride

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Abstract

The influence of the growth conditions on the properties of PTCDA thin films deposited by organic molecular beam deposition on glass substrates is presented. Powder X-ray diffraction studies show that only the α polymorph is formed, regardless of the growth conditions. By contrast, the film morphology depends specifically upon the deposition parameters, the observed crystal size increasing with growth temperature or after post-growth annealing. The spectroscopic properties (electronic absorption, fluorescence excitation and emission) are also found to be influenced by heat treatment and these are correlated with the changing crystal size in the PTCDA films. A qualitative model is presented in order to account for the photophysical properties of the films.

1. Introduction

Molecular thin films formed by vapour deposition are of significant interest for a wide range of electronic and optoelectronic device applications [1]. The perylene derivative PTCDA (perylene-3,4,9,10-tetracarboxylic dianhydride) is an archetypal molecule and its basic properties have been studied extensively during the last decade. The high crystallinity of PTCDA thin films and the low intermolecular distance in the crystal results in a strong π–π overlap and very promising transport properties [2,3]. Two polymorphic forms of PTCDA have been reported for different growth conditions, α and β, but their structures are very similar with both polymorphs crystallising in the $P2_1/c$ space group [4]. The two phases have slightly different spectroscopic properties, and have been reported to coexist when PTCDA is deposited on alkali halide substrates at high temperatures (200–260 °C) [5]. The epitaxial growth of PTCDA on a variety of substrates has also been studied extensively [6,7]. PTCDA has also been used in many practical applications such as light emitting devices, where it has been reported to improve charge injection at the anode [8], and as a hole transporting layer in organic solar cells [9]. Relatively few systematic studies on the morphological properties of PTCDA films have been carried out.

The electronic properties of PTCDA films have been studied by many research groups for growth on a variety of substrates, for different film

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thicknesses, and for variable growth conditions, although the majority of studies have been for films grown below room temperature. Surprisingly, the results are still controversial. It has long been accepted that the electronic absorption spectrum of PTCDA films originates both from monomeric transitions and excitations to a low energy state [10]. This latter state has been attributed to various species involving ionisation of the molecule, for example charge transfer [11] or Wannier–Mott [12] excitons. Theoretical studies are consistent with a mixture of Frenkel and charge transfer contributions [13,14], although the models require an increasing degree of complexity in order to account for experimental results [15]. Recent experimental studies have indicated that charge separation during optical absorption is energetically unfavourable [16], the electronic absorption spectrum being due to a superimposition of the vibronic progressions associated with monomer excited states.

In this paper, we focus on the morphological, structural and spectroscopic properties of PTCDA films grown on glass substrates at temperatures above room temperature. We find a strong correlation between the size of the crystallites and their spectroscopic properties and a qualitative model is proposed to rationalise the observed photophysics.

2. Experimental details

The films were grown on glass substrates by organic molecular beam deposition (OMBD) in an ultra-high vacuum chamber held at a base pressure of 2×10^{-10} Torr. Commercially available PTCDA powder (Fluka, 98%) was outgassed thoroughly for 72 h prior to deposition and evaporated onto the substrate from a miniature Knudsen cell. [1] The cell temperature for deposition was ~380 °C, corresponding to a growth rate of ~1 Å s^{-1}.

The film thickness was measured by a quartz crystal microbalance positioned near the substrate and verified by ex situ calibration using scanning electron microscopy studies of PTCDA films grown on silicon substrates. Typical film thicknesses range from 19 to 133 nm, although for films grown at high temperature or after annealing, partial sublimation of the material led to some reduction in the actual film thickness. For films grown at a substrate temperature >280 °C, the flux had to be increased to 1.5 Å s^{-1} to compensate for sublimation and obtain net film growth. The glass substrates were cut from microscope slides (BDH superpremium), cleaned in a methanol sonic bath for 15 min, dried by a dry nitrogen flow and transferred into the OMBD chamber. On the basis of our previous studies [17], no in situ cleaning was deemed necessary. Three growth effects were studied: (i) film thickness (19–133 nm), (ii) post-growth annealing (up to 310 °C), and (iii) deposition at high substrate temperatures (up to 310 °C).

Optical absorption data were collected using an UV/Vis spectrometer (Perkin–Elmer—Lambda 2), while the fluorescence excitation (PLE) and emission (PL) data were obtained using a fluorescence spectrometer (JYHoriba—Spex Fluoromax). A sample holder was adapted to allow illumination of the films perpendicular to the film surface. The emission was collected at an angle of approximately 20° to the incident light beam, and directed to the detector by two mirrors. Absorption, excitation and emission spectra were recorded for films grown under the different experimental conditions. Emission spectra were excited at a wavelength of 540 nm, regardless of the growth conditions. Excitation spectra were recorded at a wavelength corresponding to the emission maximum, for example 720 nm for films grown at room temperature, and 660 nm for films formed at the highest substrate temperature. Surface morphology studies were performed using an atomic force microscope (AFM, Burleigh instruments) operated in a non-contact (tapping) mode and a Nomarski optical interference microscope (Olympus BH2-UMA). The structure of the films was determined using a powder X-ray diffractometer (Siemens D5000) with a CuK$_\alpha$ source operating in the θ–2θ mode between 5 and 60°.

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[1] Photoluminescence spectra were also taken on PTCDA samples grown from twice sublimed purified powder; negligible differences were observed.

3. Results

The morphology of the films was assessed using AFM and Nomarski optical interference microscopy and was found to depend strongly on the growth conditions. Room temperature deposition produces very smooth films, which appear featureless in large area Nomarski images, Fig. 1(a). More detailed morphological information is obtained from AFM images. Fig. 2(a) shows a typical AFM image for a 133 nm thick film grown at room temperature. It is characterised by small spherical crystallites, with a typical root mean square roughness (Rq) of 4 nm. Increasing film thickness does not affect either the diameter of the spheres or the roughness of the film.

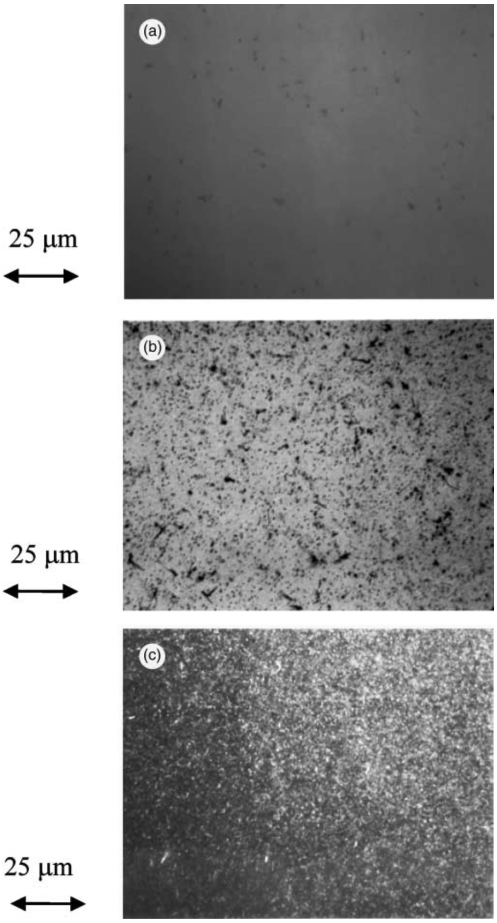

Fig. 1. Nomarski micrographs of 133 nm thick PTCDA films grown under different conditions: (a) deposition at room temperature, (b) growth at 290 °C and (c) room temperature deposition followed by annealing at 290 °C.

Growth of PTCDA films at elevated substrate temperature has a marked effect on their surface morphology. The Nomarski micrograph (Fig. 1(b)) for a film grown at 290 °C does not have the smooth, continuous appearance of the films grown at room temperature, but is instead characterised by a coarse, granular morphology. The film is much rougher (Rq ∼ 100–130 nm) and the morphology

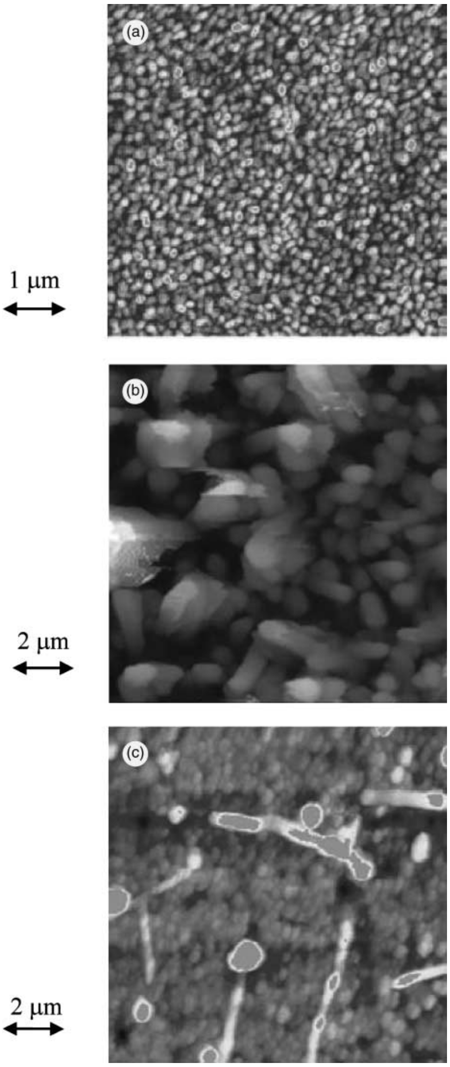

Fig. 2. AFM images of 133 nm thick PTCDA films grown at (a) room temperature, (b) a substrate temperature of 290 °C and (c) room temperature followed by annealing at 290 °C.

is dominated by elongated crystallites with a random orientation, as seen in the corresponding AFM image (Fig. 2(b)). The crystallite length ranges from 0.3 to 1 μm for growth at this temperature, but can reach 3 μm for substrate temperatures greater than 290 °C.

The morphology of PTCDA films grown at room temperature is altered significantly by post-growth annealing. The size of the spheres found at room temperature increases, typically by a factor of two, for a film annealed at 290 °C for 2 h (Fig. 2(c)). The annealed films are also characterised by a number of long, slender crystallites with a random orientation, in addition to the large spheres. These vary in size, ranging between 1–4 μm in length and 0.2–0.5 μm in width. Nomarski images of the films annealed at 290 °C (Fig. 1(c)) show randomly oriented, needlelike features superimposed on a rough underlying film. These features are up to 4 μm long and increase in size at higher annealing temperatures, consistent with the AFM observations.

The structure of the films was assessed using powder XRD. Fig. 3 shows 2θ scans for (a) a 133 nm film grown at room temperature, (b) the same film after annealing at 290 °C for 2 h, and (c) a 133 nm film grown at 290 °C. In all three scans, a single peak is observed at $2\theta \sim 27.6°$, corresponding to diffraction from the (1 0 2) plane. The lower intensity of the peak for the films prepared after heat treatment is due to partial sublimation of the material as opposed to any disordering of the crystallites, which would give rise to diffraction peaks at different 2θ values. The peak in the diffractogram is centred at 27.62° for films grown at room temperature (a), and at 27.66° for high temperature treatment (b, c). This difference is attributed to thickness and roughness differences and consequential variations in the alignment of the CuK$_\alpha$ radiation with respect to the surface, inducing a peak shift [18]. It should be noted that the full width at half maximum (FWHM) of the diffraction peak is smaller for the films prepared either at high temperature, or after annealing.

Electronic absorption spectra (Fig. 4) indicate little change in peak position or spectral shape with varying film thickness. The spectra shown in Fig. 4(a) and (b) were recorded from films of different thickness (19 and 133 nm) and both consist of a broad feature between 400 and 520 nm, and a sharp, isolated peak at ~560 nm. Annealing does not seem to have any significant effect on the basic

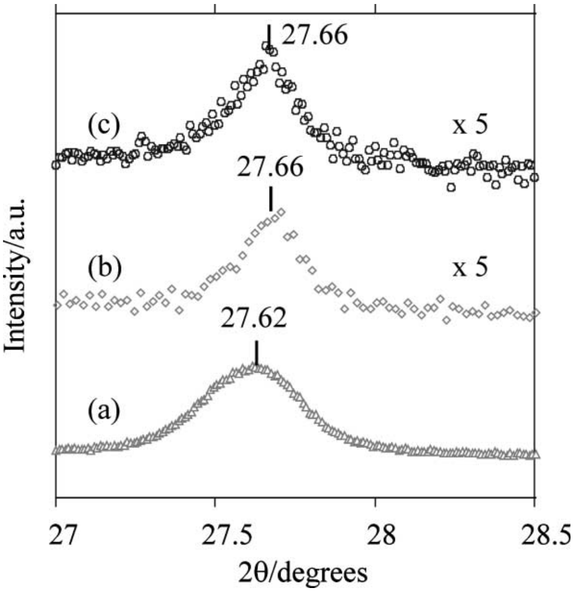

Fig. 3. X-ray diffraction 2θ scans for a 133 nm thick PTCDA film obtained at (a) room temperature, (b) after annealing at 290 °C and (c) for a substrate temperature of 290 °C. Partial sublimation and larger crystals in films that have undergone heat treatment explains the lower intensity and reduction in FWHM of the scans in (b)–(c).

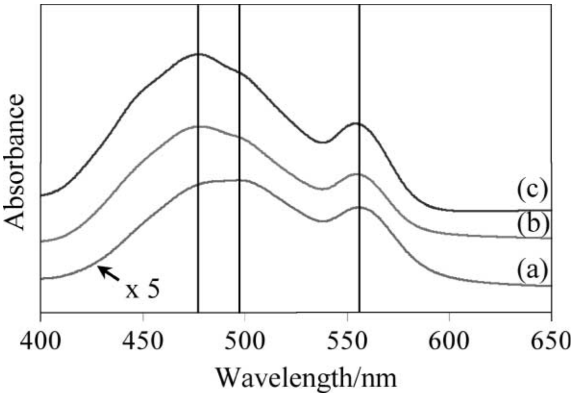

Fig. 4. Electronic absorption spectra of (a) a 19 nm thick PTCDA film deposited at room temperature and a 133 nm thick PTCDA film grown (b) at room temperature and (c) annealed at 290 °C for 2 h. The spectra are offset for clarity.

shape of the absorption spectrum, as indicated by the spectrum in Fig. 4(c), which was recorded from a 133 nm thick film that was annealed at 290 °C for 2 h. By contrast, film growth at different substrate temperatures has a marked effect on the absorption spectra. Fig. 5 shows spectra recorded from a 133 nm thick film grown at (a) room temperature, (b) 290 °C and (c) 310 °C. At higher temperatures, the short wavelength components become less prominent, and all spectral features undergo a broadening with a concomitant shift to longer wavelength.

Photoluminescence excitation (PLE) spectra provide complementary insight into the spectral properties of the films. PLE spectra are a measure of $1 - T$, where T is the sample transmission; when the absorbance (A) is small, $A \sim 1 - T$ and the PLE resembles the electronic absorption. This is verified by the PLE spectrum for a thin film grown at room temperature (Fig. 6(a)), which is very similar to the corresponding absorption spectrum. In contrast, for thicker films with a higher absorbance, the approximation fails and the PLE spectra do not correlate to the absorption spectra, as can be seen in Fig. 6(b). Films grown at elevated temperatures suffer a degree of sublimation that leads to films thinner than expected, as can be seen from the integrated absorption of the spectra shown in Fig. 5(b) and (c). The reduced absorbance should lead to a better correlation between absorption and PLE spectra, but clearly this is not the case. The shape of the PLE spectra for films grown at higher temperatures (Fig. 6(d) and (e)) are significantly different to both the corresponding absorption spectra and the PLE spectra of films grown at room temperature. The PLE shifts to longer wavelengths, with the most prominent peak occurring at the longest wavelength. Moreover, PLE spectra for high substrate temperature films can be seen to be more intense than those recorded from films grown at room temperature.

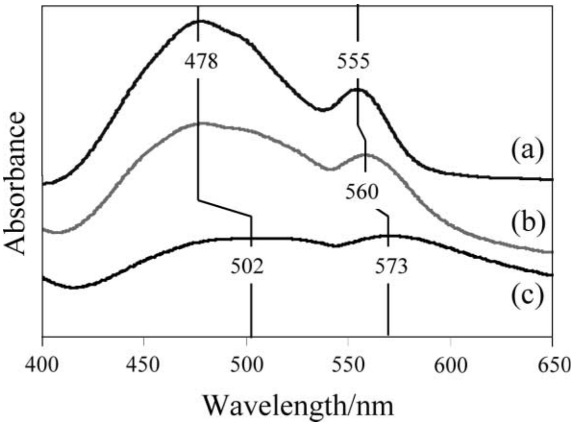

Fig. 5. Electronic absorption spectra of a 133 nm PTCDA film grown at (a) room temperature, (b) 290 °C and (c) 310 °C.

PLE spectra were also recorded for a series of emission wavelengths around the emission maximum. Fig. 7(a) displays several spectra recorded from a 19 nm thick film grown at room temperature. It is clear that the shape of the PLE spectrum does not depend on the detected emission wavelength. This independence was also observed for annealed films and, most importantly, for films grown at high temperature. Similarly, the PL emission spectra are independent of excitation wavelength, as shown in Fig. 7(b) for the 19 nm film grown at room temperature.

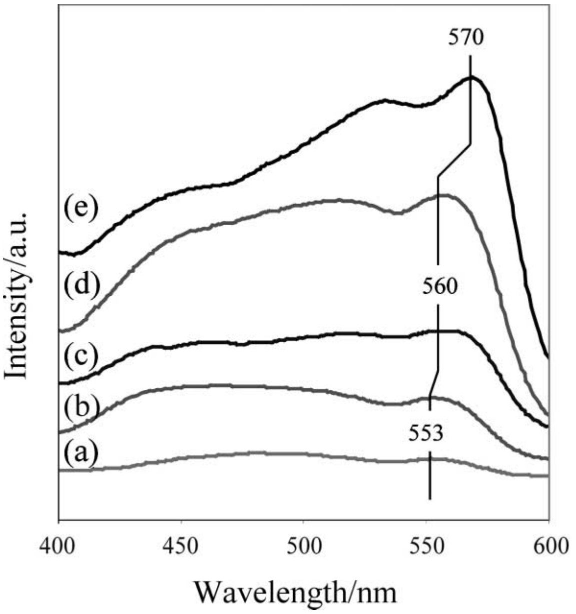

Fig. 6. PLE spectra for PTCDA films grown under different conditions: room temperature deposition with a thickness of (a) 19 nm and (b) 133 nm, (c) post-growth annealing of a 133 nm thick film and deposition of a 133 nm film with the substrate temperature at (d) 290 °C and (e) 310 °C. The spectra are offset for clarity, but intensities have not been scaled.

The PL emission spectra exhibit a strong dependence on the growth conditions. Fig. 8 shows a series of spectra recorded for films grown at room temperature (a) and (b), for annealed films (c) and (d), and for growth at elevated temperature (e) and (f). For growth at room temperature and after annealing, the emission consists of a single main broad peak at 713 nm. For the films grown at high temperature (e) and (f), the PL intensity increases significantly, in a fashion similar to that reported earlier for the PLE spectra. There is also a marked shift of the emission maximum to shorter wavelength, with the emission shifting almost 50 nm to 664 nm for the film grown at 310 °C. A shoulder also appears on the blue side of the emission spectrum at ~620 nm. It should be noted that these spectra are again independent of the excitation wavelength.

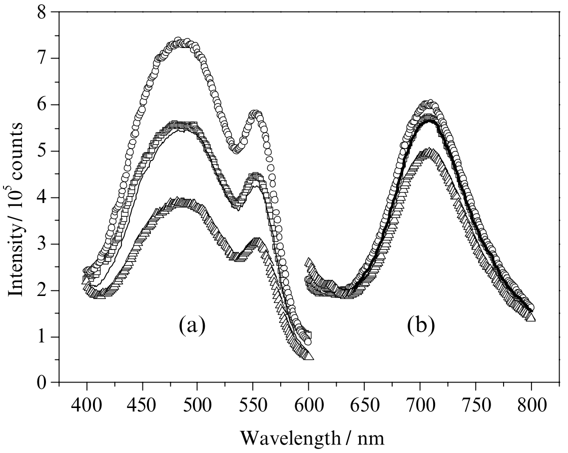

Fig. 7. (a) PLE and (b) emission spectra for a 19 nm thick PTCDA film. The excitation is measured for the emission at 680 (□), 700 (○), 740 (—) and 760 nm (△), and the emission is collected for excitations at 545 (□), 550 (○), 560 (—) and 565 nm (△).

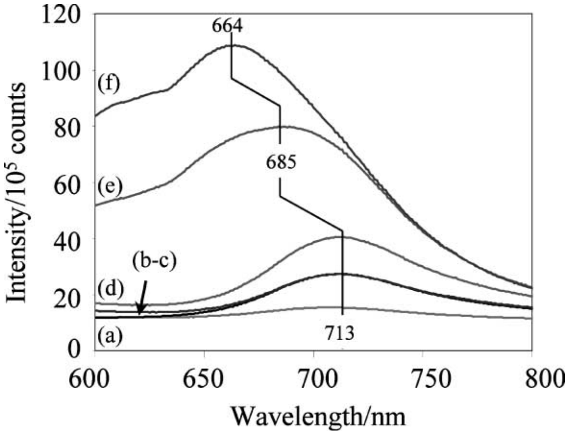

Fig. 8. PL spectra for (a) 19 nm and (b) 133 nm thick PTCDA films grown at room temperature, 133 nm films annealed at (c) 280 °C and (d) 290 °C and 133 nm films grown at a substrate temperature of (e) 290 °C and (f) 310 °C.

4. Discussion

The structural and spectroscopic results both indicate that only one polymorph is present for the set of growth conditions used. A single peak is observed in XRD at $2\theta = 27.62°$ for films grown at room temperature, and 27.66° for films undergoing high temperature treatment (Fig. 3). Previous studies by Möbus et al. [4] assigned the (1 0 2) peaks to the α and β polymorph on the basis of 2θ values at 27.67° and 27.41° respectively. We therefore identify the films as being of the α polymorphic phase for all the growth conditions studied.

Surface morphology studies (Figs. 1 and 2) show that heat treatment (during growth or annealing) leads to larger crystal sizes, although the specific details depend on the actual deposition condition. For example, larger spheres and long crystals are present after annealing, while disordered elongated crystals characterise the films grown at high substrate temperatures. The reduction in the FWHM of the XRD peak (Fig. 3) suggests that the crystal size increases with heat treatment, consistent with the surface morphology results.

The results suggest a strong correlation between the growth conditions, and both the morphology and photophysical properties of PTCDA thin films. The PLE and PL emission spectra are independent of the emission and excitation wavelengths respectively (Fig. 7), regardless of the growth conditions. This too is consistent with the structural data and indicates the presence of only one polymorph, since multiple polymorphs would give rise to wavelength dependent PLE and PL emission [5].

For all growth conditions, the electronic absorption spectra consist of a broad feature at short wavelengths, with a sharper peak at longer wavelength. Electronic absorption is not affected by the thickness of films grown at room temperature and both the shape and the peak positions are retained. When the PTCDA films are grown at elevated temperatures, the most dominant effect on the absorption spectrum is a broadening of the spectral features. The independence of the PL spectrum on excitation wavelength reduces the possibility that the broadening is due to spectral or structural inhomogeneity, but is most likely to be a consequence of strengthening intermolecular interactions [19].

The similarity of the absorption and PLE spectra for films grown at ambient temperatures, with or without annealing, are consistent with a homogeneous film, and show that emission occurs efficiently after excitation into the absorbing region. In contrast, it can be seen by comparing Figs. 5(c) and 6(e) that the PLE spectrum for films grown at high temperature is very different from the corresponding absorption spectrum, although the longest wavelength absorption feature coincides with the same feature in the PLE spectrum at 570 nm. The PLE shows a distinct vibronic progression and has a similar profile to that of molecular PTCDA in solution [11,16]. The same vibronic progression has also been shown theoretically to originate from an exciton-phonon-charge-transfer dimer [13], with the peak at 570 nm corresponding to the 0–0 transition. The difference between absorption and PLE for the films grown at elevated temperature must be interpreted with caution, and can have two origins. Firstly, the PL intensity grows significantly for these films, and it is possible that the high growth temperatures introduce a new emitting species whose emission masks that observed for films grown at ambient temperatures. Alternatively, the higher growth temperatures could be affecting the nature of the absorbing and emitting species as well as the transition between the two.

For films grown at room temperature, the large Stokes shift of 158 nm between the sharp absorption feature at 555 nm and the emission maximum at 713 nm is very marked. This is indicative of significant relaxation between the absorbing and the emitting species. It can be seen that the spectral features of absorption and emission are very different, implying that the nature of the species involved also differ. The emission spectrum shows no structural features and when plotted on an energy scale it exhibits a Gaussian profile, very similar to that predicted for an excimer (excited dimer) state. This species, by definition, exhibits no absorption and would explain the large Stokes loss, contrary to a low energy emission from a monomeric state as suggested before [20]. Since the excimer is an intrinsic trap site within the crystal, any excitation introduced either into a single PTCDA molecule or as a mobile exciton, could migrate to these sites with almost equal probability and the absorption and PLE spectra therefore coincide. Although the planar configuration of the PTCDA molecules is ideal to stabilise excimers, it is also probable that such a species might exist at a grain boundary, as observed for example in crystals of 9-cyano anthracene [19]. The consistency between the data presented here and that reported in the literature is good evidence against the emission emanating from an impurity in the sample.

The PL spectrum of films grown at high substrate temperature (Fig. 8(f)) has a reduced Stokes shift of 300 meV to the maximum emission at 664 nm. The emission shoulder at 620 nm shows an even lower shift and may be due to direct emission from the absorbing state. The emission at 664 nm may emanate from a less-stable excimer state than that observed from films grown at room temperature. It is possible that although the films appear to be structurally similar to those grown at ambient temperatures, the molecular interactions are subtly different. Indeed, molecules in larger crystals will feel more next neighbour interactions as if in smaller aggregates where the proportion of molecules on the surface (and without any neighbour) is much higher. Alternatively, the room temperature growth conditions introduce a variety of trap sites that are excimer-like, with the exciton migrating efficiently to the lowest energy site, where it subsequently emits. The high temperature growth conditions may remove the deeper traps, but the shallower ones remain and it is from these that the emission is detected. Time-resolved PL measurements will provide greater insight to this issue.

A qualitative model that takes into account these observations is shown in Fig. 9. The main absorption goes to the higher energy excited state (HEES) and the lower energy excited state (LEES), and their associated vibronic progressions. The absorption and emission data from films grown at ambient temperature do not distinguish between these two excited states. Excitation into either state results in efficient migration or relaxation to the emitting excimer states, ES. For films grown at elevated temperatures, the differences between the PLE and absorption spectra indicate that excitation into the LEES leads to more emission and a small Stokes loss. The 'LEES exciton' migrates to a different excimer state that subsequently emits at a higher energy than for films grown at room temperature. Although the figure shows a less stable excimer state, this may not be the case as the difference in energy is a combination of the excimer binding energy and the ground state repulsion term, and it is not possible to discern from the data here, which of the two effects are dominant in shifting the excimer emission to shorter wavelength.

The structured PLE spectrum for the films grown at high temperature suggests strong coupling between the electronic excitation and a molecular vibration. The origin of the electronic excitation is still the subject of some debate in the scientific community, however, it is agreed that the exciton, whether Frenkel [16] or mixed charge-transfer-Frenkel [13], couples to vibrations and produces a vibronic progression. The absorption data cannot resolve the progression readily as it falls beneath the broad absorption band at higher energy. The PLE data may therefore be the first direct evidence that this strong coupling does occur, in full agreement with theoretical models.

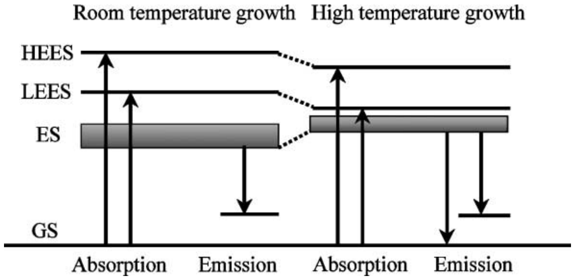

Fig. 9. Schematic of the energy levels for films grown at room temperature and at high temperature. The ground state (GS) remains unchanged, but the high energy and low energy excited states (HEES and LEES, respectively) are stabilised for high temperature films. Traps induce a number of excimeric states (ES) from which emission originates; the deep traps responsible for room temperature emission are removed by high temperature growth.

The intensity enhancement in PL emission spectra (Fig. 8) has been observed previously [21] and assigned to better crystallinity and larger particles; features that are confirmed by the AFM images shown in Fig. 2(b). The fact that the increase in PL coincides with the shift to shorter wavelength suggests that the shallow traps have a more efficient radiative decay.

5. Conclusions

The morphological and spectroscopic properties of PTCDA thin films formed by vacuum deposition are found to depend strongly on the growth conditions. The spherical crystallites that characterise films deposited at room temperature grow in size after annealing up to 310 °C and some elongated crystals are formed. A more pronounced effect is obtained for growth at high substrate temperature, where the crystal length and surface roughness are ~40 times larger than for films deposited at room temperature. The structural properties as characterised by powder XRD remain unchanged and all the films studied are of the α polymorphic form.

The films grown ambient temperatures behave in a fashion similar to the many previous reports [3,5,16]. At elevated temperatures, however, there are some very pronounced differences. The PLE spectra deviate markedly from the absorption spectra; the PL efficiency is increased and the spectrum shifts to higher energy. The large Stokes loss at ambient temperature indicates that the emission is from a different, lower-energy state than that produced by absorption, and it behaves very similar to an excimer state. It is recognised, however, that this is a simple picture and a more complex multi-molecule state is also possible. The reduction of the Stokes shift for the films grown at high temperature is good evidence for the removal of these states or the population mechanism leading to their production. The PLE data for samples grown at high substrate temperature shows fine structure that supports the vibronic origin of the absorption.

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