Film morphology and molecular orientation in oligothiophene-fluorene films

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ABSTRACT

The effects of processing conditions on film morphology and molecular orientation were studied for a novel conjugated fluorene–bithiophene oligomer, oligo(9,9-dioctylfluorene-*alt*-bithiophene) (OF8T2). Depending on the method of film preparation, OF8T2 molecules adopt different orientations in the films. X-ray diffraction peak at $4.9°$ of the OF8T2 film deposited from petroleum ether/dichloromethane mixture is attributed to a layering distance between sheets of OF8T2 chains, which are separated by the octyl side chains. Preferred orientation is clearly inferred through the absence of peaks corresponding to π–π stacking. For the spin-coated film after annealing, X-ray diffraction patterns indicate the presence of lamellar structure with the plane of the conjugated backbone normal to the substrate. The molecules were aligned with long axes along the rubbing direction when the spin-coated film was rubbed and then annealed. These results suggest a convenient approach for preparing active layers for organic optoelectronic devices by simple solution methods.

1. Introduction

Conjugated oligomers and polymers have attracted significant interests for their application as active materials in organic optoelectronic devices such as field-effect transistors (FETs) [1–3], organic solar cells [4], and light-emitting diodes (LEDs) [5,6] because of their excellent semiconducting and photophysical properties. In these devices, film morphology and orientation of the molecules in the film are critical factors for the performance of the device. For instance, it has been reported [7] that the field-effect mobility of the FET device of regio-regular poly(3-hexylthiophene) (P3HT) was up to 0.1 cm^2/Vs with the edge-on chain orientation, while the mobility with face-on orientation was 100 times lower [7].

Many efforts have been paid out for understanding and controlling polymer packing behavior in thin films [7–18]. Kim and Swager [8] synthesized poly(*p*-phenylene–ethynylene) compounds with precise structural features and were able to control the conformations of individual polymers and interpolymer interactions in Langmuir films. Yerushalmi et al. [9] found that the variation of the pyridyl nitrogen position leads to different molecular orientations in the monolayers by self-organization on chlorobenzyl-functionalized substrates. In general, to obtain different molecular orientations, in addition to the chemistry approach of altering molecular structures [8–11], changing the intermolecular

interactions at the interface between the molecules and the substrates is an important physical approach [12–15]. For example, P3HT nanocrystals can adopt two orientations, parallel or perpendicular to the insulator substrate, depending on the surface characteristics [15]. Hu et al. [16] found that in ultrathin-films the orientation of molecular chains and lamellar crystals depends on the thickness of the film. They observed that in the monolayer of poly(di-*n*-hexylsilane) (PDHS), the silicon backbones were perpendicular to the substrate surface, and in contrast, edge-on lamellar crystals with silicon backbones orienting parallel to the substrate surface grew on the sub-flat-on lamellae in films thicker than 13 nm [16].

Conjugated oligomers with well-defined structures have proved to be great models for establishing molecular structure–property relationships of various conjugated systems [19–21]. For example, poly(fluorene-*co*-bithiophene), a conjugated copolymer composed of fluorene and bithiophene units, has been considered as one of the most promising semiconducting polymers due to its excellent stability and charge transport capability [22]. To gain insights into this conjugated system, recently we synthesized a series of monodisperse oligo(fluorene-*co*-bithiophene)s and studied their adsorption and photoluminescence properties [23]. In addition, conjugated oligomers have shown potential as high-performance optoelectronic materials [24,25]. However, studies on understanding and controlling molecular orientation in oligomer thin films are very limited [12–14]. In this paper, we study the effect of processing conditions on the film morphology and molecular orientation for a fluorene-*co*-bithiophene oligomer, OF8T2 as shown in Fig. 1, [23]. A simple solution method was used to prepare

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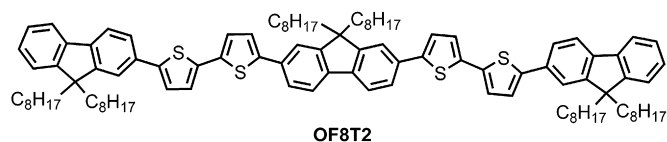 *E-mail address:* zhsu@ciac.jl.cn (Z. Su).

Fig. 1. Structure of the conjugated oligomer OF8T2.

thin films under ambient which provides orientation, and two kinds of molecular orientations in thin films can be achieved through different processing methods.

2. Experimental

2.1. Sample preparation

Prior to use, silicon wafers, cover glass plates, and mica wafers were immersed in a piranha solution (H_2SO_4–H_2O_2 70:30 v/v) at 80 °C for 2 h before thoroughly rinsed with Milli-Q water (18.2 MΩ/cm^2), and then dried in a stream of nitrogen. An OF8T2 solution in petroleum ether/dichloromethane (6:1 v/v) was prepared (concentration 1.9×10^{-4} mol/L) and stirred for different periods of time before one drop of the solution was deposited on a silicon wafer and dried under ambient. The thickness of the film was about 28 nm. In a different approach, chloroform solutions of the oligomer at concentrations ranging from 0.25 to 1.0 wt% were prepared. Films of different thicknesses were then obtained by spin-coating these solutions at 2000 rpm for 30 s. The films were then dried under vacuum for 3 h to remove the residual solvent. To achieve orientation, a film was unidirectionally rubbed with a velvet cloth in a homemade machine (velocity 5 mm/min, pressure 58 g/cm^2).

2.2. Measurements

X-ray diffraction data were collected on a D/max 2500PC diffractometer equipped with a Rigaku 18 kW rotating-anodes generator (Cu Kα) operated at 50 kV and 250 mA with a scanning rate of 2°/min. The 2θ angle ranged from 3° to 30°. Tapping-mode atomic force microscopy was performed on a Seiko SPA-300HV microscope with an SPI 3800N controller. Etched silicon cantilevers with a spring constant of 42 N/m were used. A Leica polarized optical microscope (POM) was employed for optical microscopic studies. A Perkin–Elmer Lambda 900 UV/VIS/NIR spectrometer equipped with a polarizer was used for optical measurements.

3. Results and discussion

3.1. Morphology of films deposited from petroleum ether/ dichloromethane mixture

OF8T2 is a typical π-conjugated hairy-rod molecule. For hairy-rod molecules, different chemical nature of the stiff backbone and the flexible side chain can lead to formation of interesting microstructures. The addition of petroleum ether (a poor solvent) provides an environment for self-organization and molecular aggregation of OF8T2. The best results were obtained when the petroleum ether/dichloromethane ratio was 6:1 (v/v) at the OF8T2 concentration of 1.9×10^{-4} mol/L, where the solution was just saturated. Fig. 2 shows the AFM height images of OF8T2 films cast from the petroleum ether/dichloromethane solution on silicon wafers after stirred prior to casting for 1, 24, 48, and 72 h, respectively. Needle-like crystals about 70–100 nm in width and 1 μm in length were observed for the solution stirred for 1 h (Fig. 2a). Some of them were connected and were the coalescence of two or more

individual crystallites. This morphology appeared throughout the whole sample on different substrates used, including silicon wafer, cover glass plate, and mica wafer. From the topographic image the surface roughness of the film was measured to be about 2.1 nm. For the film cast from the solution stirred for 24 h, the domain size extended to 100–200 nm in width and 1–2 μm in length (Fig. 2b), and connected needle-like crystals still remained. For the film cast after 48 h and longer stirring, the crystals became larger and flat with poor long-range continuity (Fig. 2c and d). Furthermore, in the solution standing for several days yellow crystals of several millimeters long were observed to precipitate. These crystals of different sizes obtained at different stirring times and on various substrates all have same crystal structure as indicated by XRD (discussed in next section), suggesting that the unique molecular packing in these crystals is due to the architecture of the oligomer.

In a solution, the solvent quality for the backbone and for the branch chains can be judged by the interaction parameters (χ_{bb-s} and χ_{br-s}, respectively). The interaction parameter for the backbone, χ_{bb-s}, is evaluated by the following equation [26].

$$\chi_{bb-s} = V_s(\delta_s - \delta_{bb})^2/RT + 0.34$$

where V_s is the molar volume of the solvent, and δ_s and δ_{bb} are the solubility parameters for the solvent and the backbone, respectively. The interaction parameter for the branches is calculated in the same way. In our case, petroleum ether is a good solvent for the alkyl side chain ($\chi_{br-s} < 0.5$), and a poor solvent for the OF8T2 backbone ($\chi_{bb-s} \gg 0.5$) [27]. The compatibility difference between the stiff rods and the flexible side chains with the petroleum ether acts as the driving force for the intermolecular self-assembly. In addition the OF8T2 molecules tend to aggregate in a face-to-face packing mode into rod-like particles due to the π–π interactions between the conjugated segments [28,29], which can decrease the unfavorable interactions between the solvent and the aromatic backbone units [29]. Molecular modeling simulations also support the face-to-face packing mode of the OF8T2 molecules [30]. The most stable configuration for the stacks is that the bithiophene units of one chain lie in front of the fluorene units of the adjacent chain with the octyl side chains extending in a quasi-perpendicular orientation relative to the conjugated chain [30]. Favorable π–π interaction induces more oligomer chains in the solution to join the stacks, resulting in the growth of the crystals. As seen in Fig. 2, the size of the particles increases dramatically with the time the solution is stirred. The process is probably controlled by the solvent evaporation kinetics. Compared with petroleum ether, dichloromethane, the good solvent for the OF8T2, has a lower boiling point and is more volatile. So with the stirring time increasing, more dichloromethane evaporates, leading to both the increase of the OF8T2 concentration and the deterioration of the solvent quality (increase of the fraction of the nonsolvent, petroleum ether). These are the two key factors governing the nonequilibrium state of the aggregation process before deposition. Molecular aggregation continues to happen in the solution [31,32], and the longer the stirring time, the bigger the crystals grow in the solution before deposition. Finally after several days, yellow crystals of several millimeters long can precipitate from the solution, which is an obvious evidence for the existence of crystallization in the solution before casting.

Besides the stirring time, the rate of solvent evaporation also affects the shape of the crystals when the stirring time is the same. The length of the needle-like crystals increased from 1 to 2 μm to several micrometers by slow solvent evaporation in the presence of a solvent vapor pressure because second aggregations inside droplets occurred during the solidification process [28]. Therefore when we investigated the effect of the stirring time on the particle
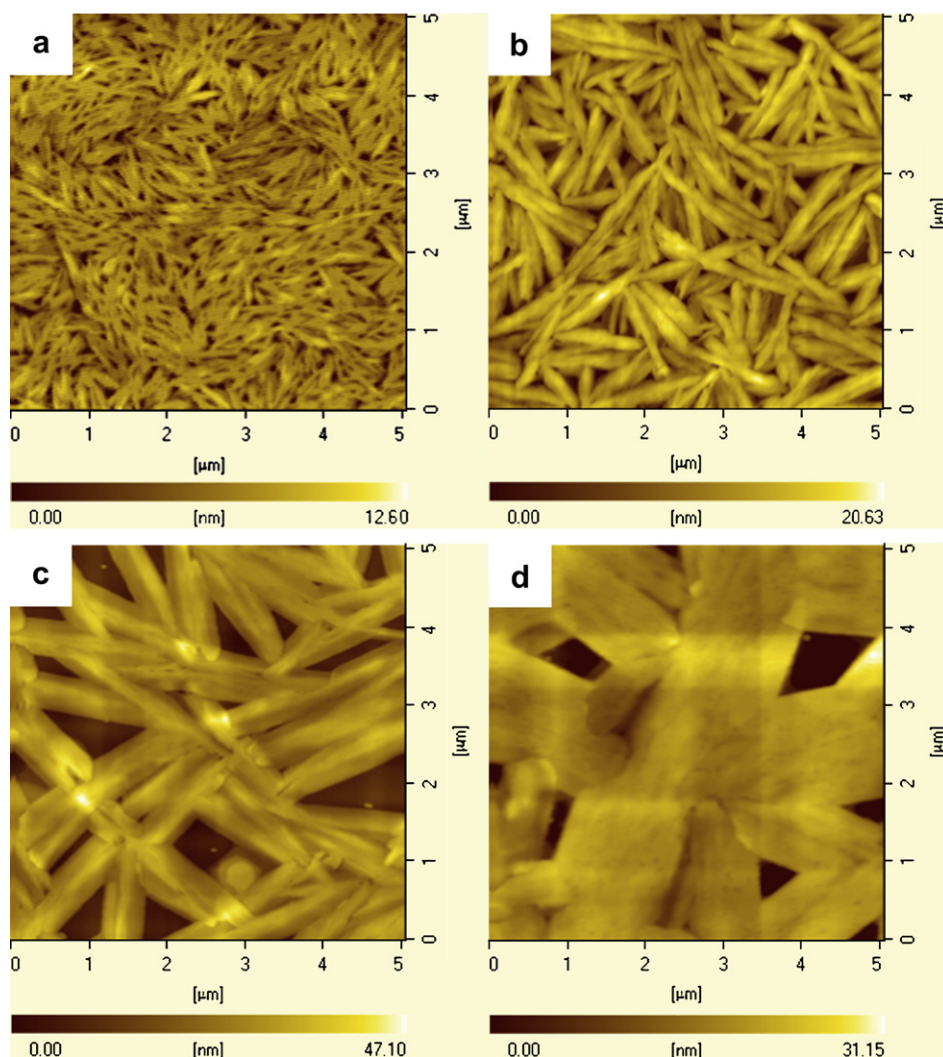
Fig. 2. AFM topographic images of OF8T2 films on wafers deposited from petroleum ether/dichloromethane mixture (6:1 v/v) after stirred for (a) 1, (b) 24, (c) 48, and (d) 72 h.

size, relative fast solvent evaporation was applied for fast forming of the film to preserve the pristine morphology of the crystals in solution by suppressing the secondary aggregations inside the droplets, which would occur upon slow evaporation of the solvent.
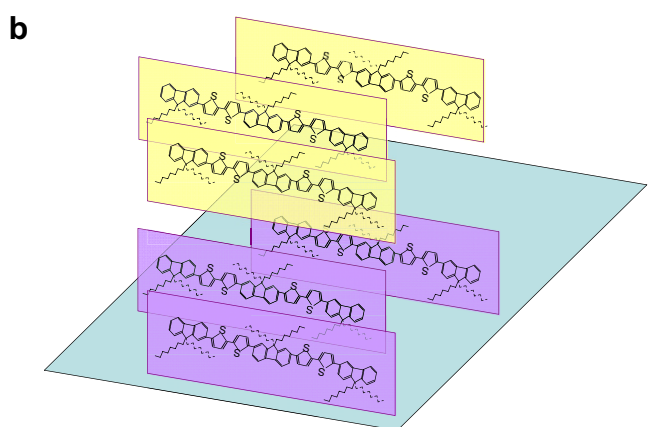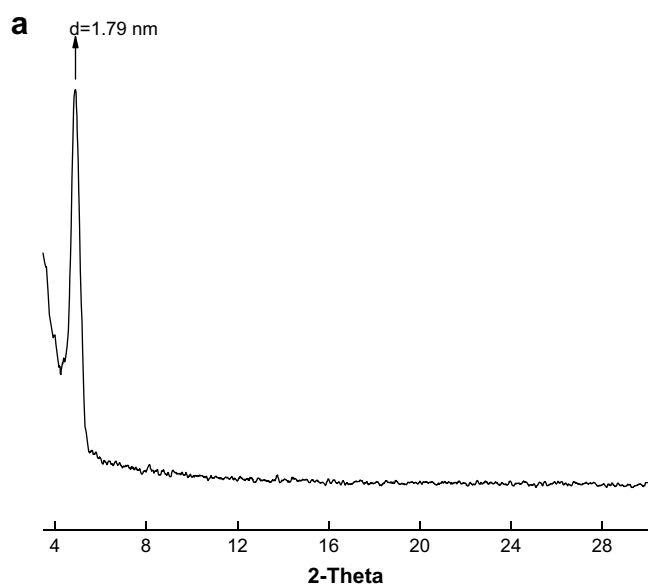
3.2. Structure of the film deposited from petroleum ether/dichloromethane mixture

The X-ray diffraction patterns of all these films were similar, and a typical example is shown in Fig. 3. The peak at $2\theta = 4.9°$ is attributed to a layering distance (d_{100}) of 1.79 nm between sheets of OF8T2 chains separated by the octyl side chains orienting normal to the aromatic planes [33,34]. The 1.79 nm distance is consistent with the total size of the plane of backbone and the octyl groups in an extended configuration. Preferred orientation is clearly inferred through the presence of this peak and the absence of peaks at about 0.3–0.5 nm corresponding to π–π stacking (see Fig. 3b) [35]. The alignment of the oligomer chains is important to enhance the carrier transfer property in the oligomer FETs. Although compared with poly(3-hexylthiophene), the presence of two octyl side groups on the fluorene units increases the steric hindrance between two adjacent OF8T2 chains; preferred orientation is still achieved by this simple method for the following reasons.

When the solution is deposited on silicon wafer, the arrangement of the OF8T2 molecules is governed by the shape effect, which is the predominant factor. These needle-like crystals are about 70–200 nm wide, 1–2 μm long, and only about 5–30 nm thick, i.e. the length is about 10 times greater than the width, which is in turn about 10 times greater than the thickness. When the solution is deposited, the needle-like crystals in the solution under the influence of gravity would most probably lie flat on the substrate because of their large aspect ratios, resulting in the oligomer molecules orienting with the octyl groups normal to the substrate. This model of aggregation is consistent with the X-ray diffraction data.
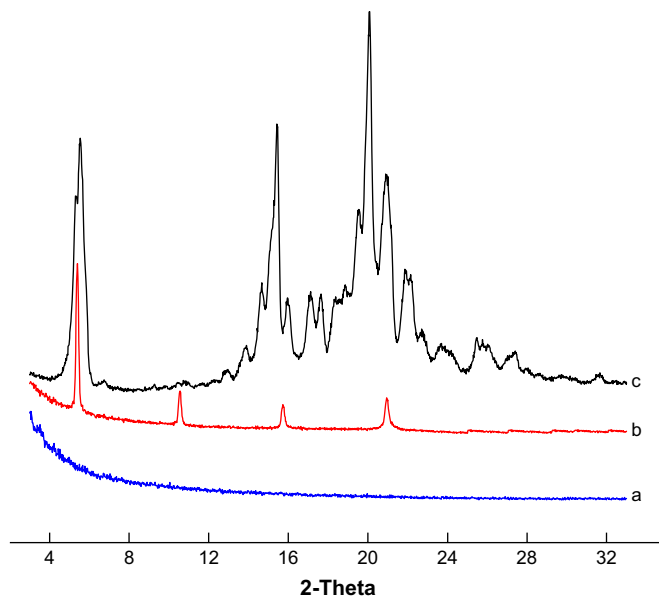
3.3. Structure of the spin-coated film

We also prepared OF8T2 films by conventional spin-coating. Wide-angle X-ray diffraction (WAXD) was then applied to investigate the structure of the films, and the diffraction patterns are shown in Fig. 4. The diffraction pattern of the OF8T2 powder is also included as a comparison, where many peaks are clearly observed. First it can be seen that the as-cast thin film exhibits no diffraction peaks, indicating the absence of any crystalline structure, which is consistent with literature report [36] that chains are randomly oriented in spin-cast films. Upon annealing, the randomly oriented molecules begin to reorganize, and four sharp diffraction peaks are

a

d=1.79 nm

2-Theta

b

Fig. 3. X-ray diffraction pattern of the needle-like crystals obtained by drying a drop of OF8T2 solution in petroleum ether/dichloromethane mixture (6:1 v/v) after stirred for 24 h (a), and a schematic representation of the molecular packing in the film (b).

2-Theta

Fig. 4. X-ray diffraction patterns of a spin-coated OF8T2 film as-cast (a), and after annealed at 75 °C for 1 h (b); (c) the diffraction pattern of OF8T2 powder.

third-, and forth-order diffractions observed for the spin-coated film were absent in the diffraction pattern of the needle-like crystallites.

3.4. Morphology and molecular orientation in rubbed film

Molecular alignment has proved to be very important in many device applications. The properties of oriented materials can be improved by orders of magnitude. It is well known that spin-cast process results in the flexible polymer chains randomly oriented within the film because of the formation of the entanglement network on the substrate. For rigid-rod polymers only a small number of the chains are aligned or partially aligned in the radial direction [38b]. To align the π-conjugated hairy-rod molecules, the thin films were unidirectionally rubbed with a velvet cloth on a homemade machine. Fig. 5a is the optical micrograph of the film observed under crossed polarizer. It was found that the light intensity was maximum when the rubbing direction formed a 45° angle with the polarizers, and complete extinction was observed when the incident light polarized either parallel or perpendicular to the rubbing direction (Fig. 5a, inset). The observation indicates that the OF8T2 molecules aligned along the rubbing direction. A shear stress upon rubbing induced plastic deformations and grooves parallel to the sliding direction appeared. When the films were heated under N_2, the grooves vanished at 60 °C because of relaxation, and then small domains came out along the rubbing direction (Fig. 5b). Our DSC analysis indicated that the glass transition temperature (T_g) of the OF8T2 is 37 °C and the melting endotherm is at ∼128 °C (data not shown). In addition, for a spin-cast film going through the same heating process, spherulitical morphology was observed by POM (Fig. 5c). Based on these observations, we conclude that the small domains are the aggregates of crystallites. The orientation induced by rubbing is the result of the surface stress, which doesn't completely relax during heating, and is preserved through crystallization [40]. The crystalline aggregates propagate the orientation from the surface to the whole film by acting as nuclei [40].

The degree of orientation was then estimated by polarized UV–vis absorption spectroscopy (Fig. 5d). The absorption maxima appear at 448 and 176 nm in the spectral region of π–π* transition. The dichroic ratio ($R = A_\parallel/A_\perp$) for light polarized parallel vs

observed at 5.22°, 10.44°, 15.67°, and 20.90°, respectively. These peaks correspond to the first-, second-, third-, and forth-order diffractions arising from the layering distance between sheets of the main chains separated by the octyl groups orienting normal to the substrate [33,34,37]. The other diffraction peaks observed for the powder are absent. This result indicates that the OF8T2 molecules arrange in the thin films with the plane of conjugation normal to the substrate. It is known that spin-coating process results in the molecular chains lying in the film [38], and the molecular motion in the thin film is limited due to spatial constraint and surface constraint [39]. Without any specific interaction at the chain ends, the standing up of the stiff molecules on their ends is very unlikely.

Compared with the diffraction pattern shown in Fig. 3a for needle-like crystallites, where only one peak is observed at 4.9° with a full width at half-maximum (FWHM) of 0.34° corresponding to a layering distance of 1.79 nm, here the diffraction for the layering distance is at 5.22° (1.69 nm) and the FWHM is 0.17°. The slightly smaller layering distance and the significantly narrower peak width indicate that the crystallites in the annealed spin-coated film were more compact and perfect than the needle-like crystallites deposited from the mixture solvent of petroleum ether and dichloromethane, where some solvent molecules might be incorporated in the lattice. This also explains why the second-,
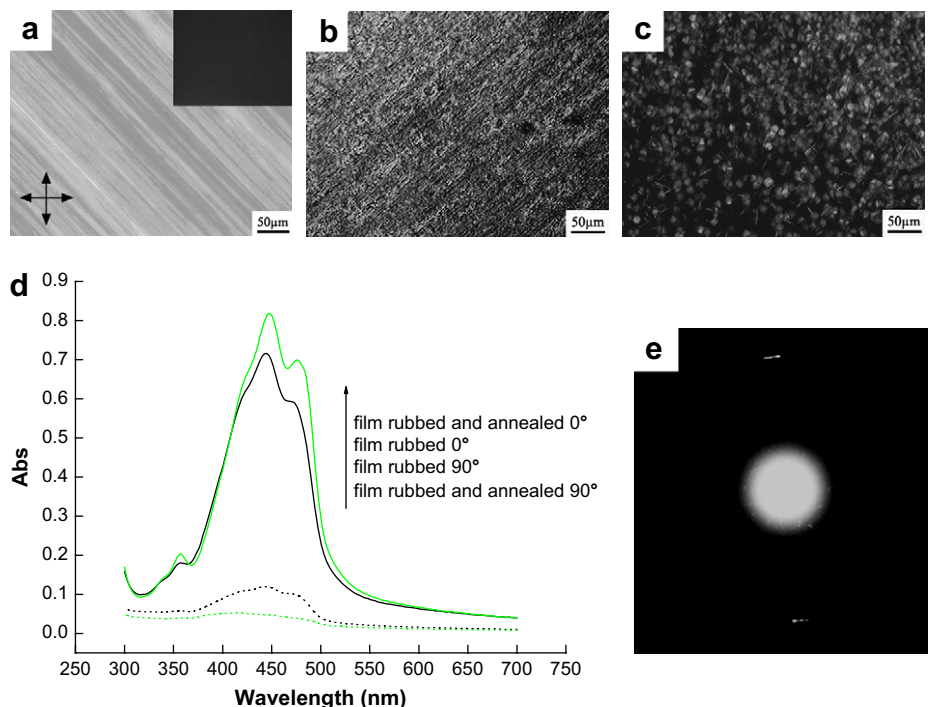
Fig. 5. (a) POM image of a spin-coated film rubbed with a velvet cloth taken under crossed polarizers with the alignment director forming a 45° angle with the polarizers, and the inset shows the film arranged with the rubbing direction parallel or perpendicular to the director of the first polarizer. (b) POM image of the rubbed film annealed at 75 °C. (c) POM image of a spin-coated film annealed at 75 °C. (d) Polarized UV–vis absorption spectra of a OF8T2 thin film rubbed (black) and then annealed at 75 °C (green) parallel (solid) and perpendicular (dotted) to the rubbing direction. A polarizer angle of 0° is consistent with the rubbing direction. (e) Electron diffraction pattern of a rubbed and crystallized OF8T2 thin film. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

perpendicular to the rubbing direction is 6.0, which reflects that the transition dipole moment is oriented predominantly parallel to the rubbing direction. The dichroic ratio of the rubbed film was further increased by several times after annealed at 75 °C for 1 h. A dichroic ratio of 17.2 was achieved. As a comparison, the literature values reported for the polymer PF8T2 are 5–12 [22b,33]. This result suggests that the chains tend to orient further with the crystallization process during heat treatment, i.e. the aligned OF8T2 crystallites act as nuclei and induce more molecules to crystallize along the same direction upon annealing, leading to a higher degree of orientation. Electron diffraction pattern of the rubbed film is shown in Fig. 5e, and the presence of arcs reveals that the oligomer molecules are aligned along the rubbing direction. The distance of 0.45 nm arises from the π–π stacking, and is attributed to the lateral distance between oligomer chains within the layer [33,34,37], which further confirms that the OF8T2 molecules arrange in the thin films with the plane of the conjugated normal to the substrate. The lateral distance is larger between two adjacent OF8T2 chains than that reported for P3HT [41] because the presence of two octyl side groups on the fluorene units increases the steric hindrance between two adjacent OF8T2 chains.

3.5. Influence of temperature on the film deposited from mixture solvent

For the OF8T2 film obtained by casting from the mixed solvent of petroleum ether and dichloromethane discussed above, it would be interesting to explore that how the molecular packing is affected by thermal treatment, and this was studied using in situ X-ray diffraction. A film cast from the mixed solvent stirred for 24 h, the morphology of which is shown in Fig. 1b, was heated at a rate of 10 °C/min, and the XRD profiles at different temperatures are shown in Fig. 6. The peak observed at 4.9° for the pristine film shifts to 5.2°, and three new peaks centered at 10.44°, 15.67°, and 20.90°,
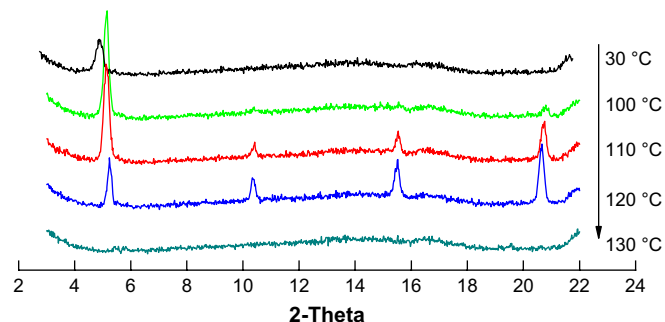
Fig. 6. Temperature-resolved XRD profiles of an OF8T2 film prepared from petroleum ether/dichloromethane mixture (6:1 v/v) at different temperatures.

respectively, appear as the temperature increases to 110 °C. This pattern matches that for the spin-cast films after annealing, which corresponds to the lamellar structure as discussed above for the spin-cast films. The structure change may be attributed to loss of the solvent molecules incorporated in the lattice upon heating, which results in a smaller layering distance and more compact and perfect crystallites. All peaks disappear at above 130 °C, indicating that the crystallites melt and the film is in an isotropic state, consistent with our DSC result.

4. Conclusions

For a hairy-rod molecule such as oligo(9,9′-dioctylfluorene-*alt*-bithiophene) (OF8T2), the processing conditions play an important role in determining the orientation of the molecules in thin films and also affect the film morphology. By using a mixed solvent and taking advantage of the solubility difference between the side chains and the backbone, the OF8T2 molecules can self-assemble and crystallize in the solution, and films cast from this solution

exhibit a preferred molecular orientation with the plane of the backbone being perpendicular to the substrate. On the other hand, lamellar structure occurs in the annealed spin-cast films with the plane of the conjugated backbone normal to the substrate with smaller layering distance and more perfect packing. Further more, unidirectional alignment of the molecules can be induced by rubbing the spin-cast film, and after annealing, a dichroic ratio of 17.2 measured by polarized UV-vis spectroscopy demonstrates a high degree of alignment along the rubbing direction. This work may provide a convenient approach for preparing active layers for organic optoelectronic devices by simple solution methods.

Acknowledgment

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