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P3HT STRIPE STRUCTURE WITH ORIENTED NANOFIBRILS ENABLED BY CONTROLLED INCLINING EVAPORATION*

Xiang Gao and Yan-chun Han**

State Key Laboratory of Polymer Physics and Chemistry, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences; Graduate School of the Chinese Academy of Sciences, Changchun 130022, China

Abstract The preparation of the poly(3-hexylthiophene) (P3HT) stripe structure with oriented nanofibrils prepared by controlled inclining evaporative technique is reported. The distance of the adjacent stripes could be controlled from 40 μm to 100 μm by decreasing the inclining angle. The oriented nanofibrils in the stripes can be obtained because the P3HT lamellae diffuse directionally and form 1D crystals at the three-phase contact line of the drop. In order to get the oriented P3HT stripes, the proper solvent evaporation rate which is controlled by the inclining angle and the wettability of the substrate must be carefully chosen to match the P3HT 1D crystallization rate. It is found that large inclining angle and the hydrophilic substrate (for example: glass and PEDOT) are beneficial to get P3HT stripe structure with oriented nanofibrils.

Keywords: Inclining evaporative technique; Orientation; Stripe structure.

INTRODUCTION

Regioregular poly(3-hexylthiophene) (P3HT) has attracted great interest for the applications in organic field effect transistors (OFET) and solar cells, because it has good electrical properties[1], chemical stability and easy processability[2]. Its rigid backbone with a regular head-to-tail arrangement of pendant hexyl side chains allows the π-π stacking of the conjugated backbones[3–10]. This 1D self-organization of π-conjugated polymers on substrates offers the strategy for the stable nanoscale structures which are dramatically influenced by regioregularity[11], molecular weight[12–16], solvent properties[13, 17] and processing conditions[17]. To get the high field-effect mobility, the film should have high crystallinity and good orientation[18, 19].

The conjugated polymer ordered structure can be obtained by mechanical rubbing of the polymer[20, 21], friction-transfer[22], deposition of a solution using directional casting[23] or using of an orienting substrate[24]. Several of these methods have been tested on P3ATs. It is reported that the ordered structure could be formed from an evaporation process[25–35]. In the process, nonvolatile solutes in the sessile droplet are ready to assemble into 1D or 2D ordered structures. Concentric rings often form due to the repeated microscopic pinning and depinning events (*i.e.*, "stick-slip" motion)[25, 26] of the three-phase contact line. Unfortunately, the concentric rings formed are generally random. By now, several elegant approaches[29–35] have been successfully exploited to control the droplet evaporation in confined geometries. Constraining a drop of polymer solution in a confined geometry composed of a curved upper surface on a flat substrate leads to a capillary-held solution. Because of the geometrical constraint imposed, the evaporation is restricted to occur only at the capillary edge[27, 28]. As a

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** Corresponding author: Yan-chun Han (韩艳春), E-mail: ychan@ciac.jl.cn
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result, the repetitive pinning and depinning of the three-phase contact line controlled by geometry constraint yields a lateral surface morphology naturally and spontaneously that consists of hundreds of highly ordered concentric polymer deposits with a regular spacing. Other ordered yet complex structures including spokes, fingers, serpentines and hierarchically organized structures can also be produced[27–35].

However, with the evaporation of the droplet, the changing curvature radius of the capillary bridge altered the instability condition between pinning and depinning of the contact line that induced the changing of the evaporation rate of the solvent. Due to the uncontrollable evaporation rate, the P3HT molecules could not form the oriented structures in the pattern. In this study, we used the method called the inclining evaporation approach, *i.e.* the P3HT drop was evaporated on an inclining substrate, different from the confined geometries used to get the P3HT stripe structure reported previously. The oriented nanofibrils in the stripe structure could be obtained by controlling the inclining angle and the hydrophilic substrate.

EXPERIMENTAL

Materials

The regioregular poly(3-hexylthiophene) (98.5% head-to-tail regiospecific conformation, M_n = 68 kDa) was purchased from Sigma-Aldrich Co. The solvent, toluene, was purchased from Beijing chemical plant and used as received.

The glass substrate: The glass substrates (1 cm wide and 5 cm length) were cleaned in a Piranha solution (70/30 *V*/*V* of concentrated H_2SO_4 and 30% H_2O_2) at 90 °C for 20 min, thoroughly rinsed with deionized water, and finally blown dry in nitrogen.

The PEDOT substrate: The glass substrates were cleaned then dried at 110 °C for 40 min. 40 nm thick poly(ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS, Baytron P4083) layer was spin-coated on the ITO glass and dried at 120 °C for 30 min.

The OTS substrate: The cleaned glass substrates were immersed in a 0.1% (*V*/*V*) solution of OTS in *n*-hexane. After 5 min they were removed from the OTS solution, washed with *n*-hexane for several times, and quickly dried under a N_2 flow.

Sample Preparation

P3HT was dissolved in the solvent with concentration of 0.4 mg/mL. First, the P3HT solution was dissolved in solvent and placed at elevated temperature 80 °C for complete dissolution. After cooling to room temperature, the solutions were standing in dark and vibrationless environment over two days at room temperature to obtain homogeneous samples.

The inclining substrates used were the glass, PEDOT, or OTS substrates as mentioned. One side of the wide edge was put to contact to the horizon plane, the other side was put to the gasket. The inclining angle was the angle between the inclining substrate and the horizontal plane. It could be controlled by adjusting the height of the gasket.

Characterization

The surface morphology was characterized by atomic force microscopy (AFM) and transmission electron microscopy (TEM). A commercial atomic force microscope in tapping mode (SPA300HV with a SPI3800N Probe Station, Seiko Instruments Inc., Japan) by using a silicon cantilever (nominal spring constant 2 N/m and resonant frequency *ca*. 70 kHz, Olympus, Japan) was used to investigate the morphological features in the polymer blend thin films.

TEM was performed on a JEOL JEM-1011 transmission electron microscope under 100 kV. Thin films were first floated on deionized water, and then transferred onto copper grids. The samples were dried at room temperature for 24 h before TEM experiments.

The grazing incidence X-ray diffraction (GIXD) profiles were obtained by using a Bruker D8 Discover Reflector with X-ray generation power of 40 kV tube voltages and 40 mA tube current. The measurements were obtained in a scanning interval of 2θ between 4° and 30°. To increase GIXD peak intensity for investigating the

crystallinity and orientation that prevail throughout the film, we employed an incident angle (α = 0.2°) slightly above the critical angle (α_c = 0.18°).

UV-Vis absorption spectra were acquired on a Lambda 750 spectrometer (Perkin-Elmer, Wellesley, MA) with 5.0 nm slit width.

The orientation of the P3HT films was proven by cross-polarized optical microscopy (C-POM) (Carl Zeiss A1m microscope) and polarized UV-Vis (P-UV-Vis) absorption spectroscopy. (C-POM) was used to record the brightness of the films at different angles. To calculate the order parameter of P3HT films quantitatively, P-UV-Vis absorption spectroscopy was also recorded with a Lambda 750 spectrometer (Perkin-Elmer, Wellesley, MA) equipped with a green prism.

As we know, the dipole vector for the light absorption of a conjugated polymer is parallel to the plane of the conjugated ring. Because of the parallel alignment of P3HT chains, the oriented film with parallel (p) polarization showed a maximum absorbance; in contrast, with orthogonal (o) polarization, the corresponding absorption intensity was much weaker. From the two curves, the dichroic ratio D can be calculated using the following equation:

$$D = A_p/A_o \quad (1)$$

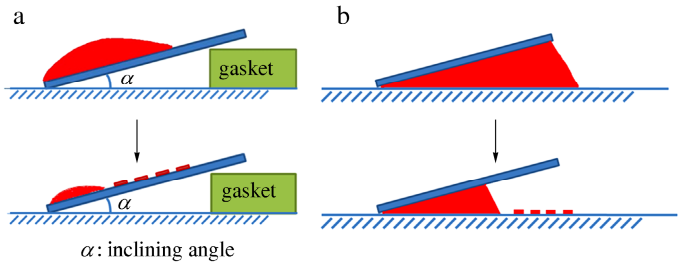
A_p and A_o are the maximum absorption intensities for light polarized parallel and orthogonal to the polymer chain orientation, respectively. The dichroic ratio D quantifies the degree of alignment by comparing the absorption polarized parallel to the polymer chains and perpendicular to them. When D value is large, the anisotropic alignment is good.

RESULTS

In our experiment, the oriented P3HT stripe structure was prepared by controlled inclining evaporative technique. During the film-formation process, two intrinsic parameters should be controlled to form the oriented structure: (1) the inclining angle of the substrate and (2) the wettability of the substrate.

Effect of the Inclining Angle of the Substrate on the P3HT Stripe Structure

As shown in Scheme 1(a), 40 μL P3HT toluene solutions were loaded onto the inclining substrate. The drop was trapped by the substrate's edge. The surface tension-held P3HT toluene solution was formed, with the highest evaporation rate at the edge of the drop, but only the three-phase contact line in the upper side can move because of the gravity. This geometry led to a controlled, repetitive ''stick-slip'' motion of the three-phase contact line, which moved towards the bottom edge of the substrate during the evaporation of toluene. As a result, the oriented stripe structured microscopic patterns formed in the inclining substrate.



Scheme 1 Schematic illustration of (a) the inclining substrateand and (b) the droplet evaporation from a restricted geometry

It could be found that the distance of the adjacent stripes and the orientation of the film will be affected by the inclining angle.

Optical images of the film produced with different inclining angles are shown in Fig. 1(A). When the angle was 5.7°, the distance was about 100 μm. With the angle increasing, the distance of the adjacent stripes became smaller. When the angle was 11.3°, the distance was about 40 μm.

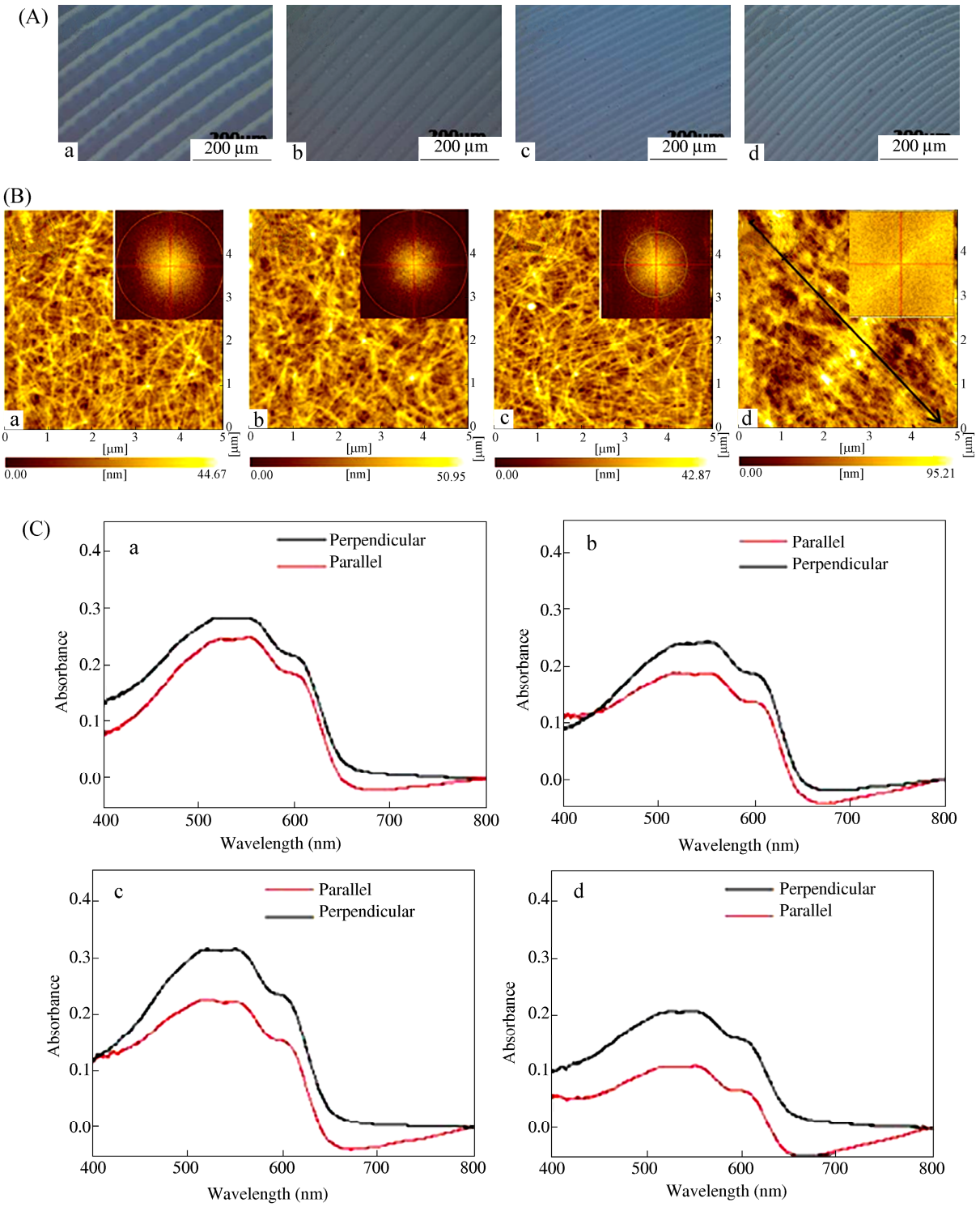


Fig. 1 Optical images (A), AFM height images (B) and UV-Vis absorption spectra using polarized light parallel and perpendicular to the direction of the aligned P3HT microfibrils (C) for films prepared with different inclining angle of (a) 2.9°, (b) 5.3°, (c) 8.9° and (d) 11.3°

The degree of the orientation of the film was increased with increasing the inclining angle. In Fig. 1(B), the orientation of the P3HT nanofibrils became better with the increasing of the angle. The fast Fourier transform (FFT) image could confirm the results. When the inclining angle is 2.9°, it could hardly find the orientation in the FFT result. However, the FFT image could confirm the orientation when the angle is 11.3°. When the angle is larger than 11.3°, the surface tension cannot hold the P3HT solution drop. The UV-Vis absorption spectra could confirm that the orientation would become better with increasing of the angle. As shown in Fig. 1(C), the dichroic ratio *D* according to Eq. (1) became larger with the increasing of the angle. It was changed from 1.14 to 1.89 when the angle was increased from 2.9° to 11.3°.

Effect of the Wettability of the Substrate on the P3HT Stripe

It is found that the hydrophilic substrate is beneficial to get the anisotropic P3HT stripe structure. By using the hydrophobic substrate, the P3HT stripes were not oriented. Three different substrates, glass, PEDOT and OTS, were used to make the P3HT stripe structure. As shown in Fig. 2(a), when the substrate was glass, and the inclining angle was 11.3°, the optical anisotropic P3HT stripes could be obtained. Under cross-polarized light,

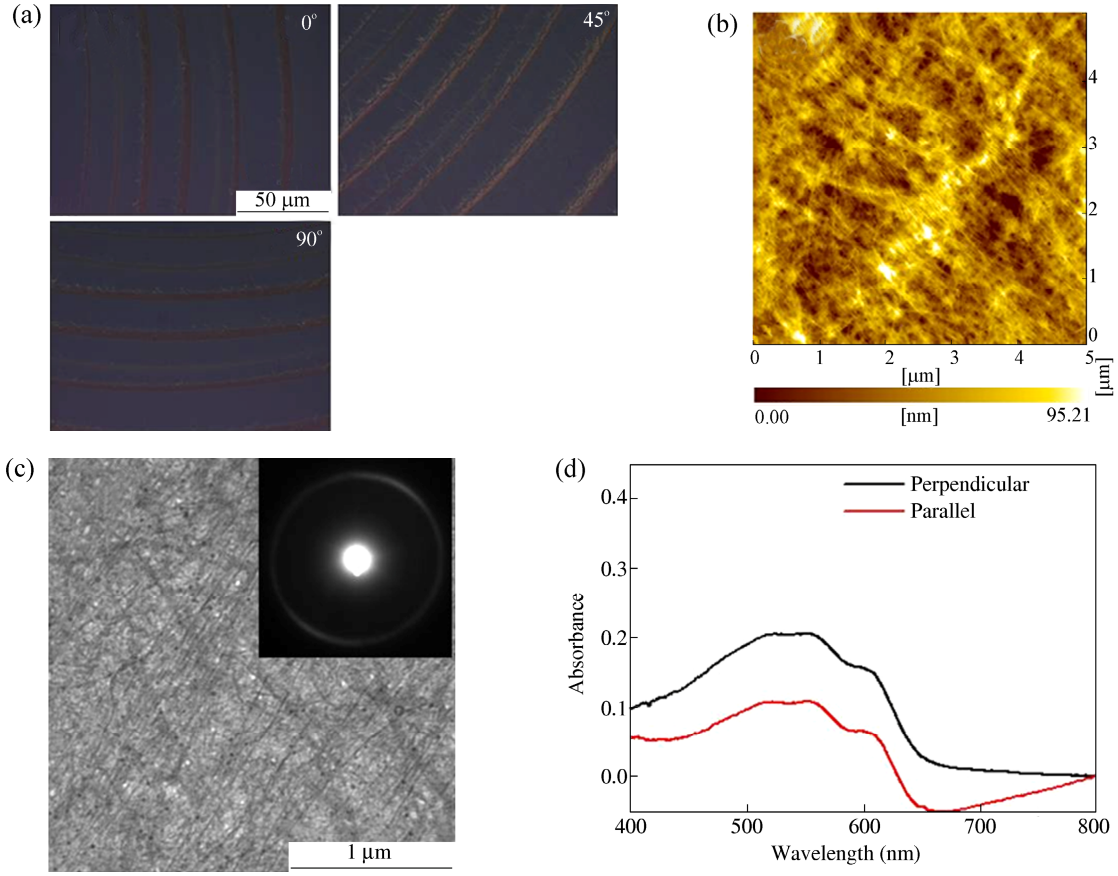


Fig. 2 The oriented zone of the P3HT film made by inclining glass substrate with an angle of 11.3° (a) optical images and C-POM recorded over the same area at different angles (0°, 45° and 90°) between polarized light and the nanofibril orientation obtained by rotating the sample, (b) AFM height image of the anisotropic film, (c) TEM image of the anisotropic film and its corresponding SAED pattern and (d) UV-Vis absorption spectra using polarized light parallel and perpendicular to the direction of the aligned P3HT nanofibrils

when the sample was rotated from 45° to 135° with respect to the direction of polarized light, the stripes presented a bright-dark-bright variation, which implied that the P3HT nanofibrils in these regions aligned parallel and then resulted in optical anisotropy. The AFM height image in Fig. 2(b) shows that the P3HT nanofibrils are perpendicular to the stripe. Because of the flexibility of long fibrils, the fibrils cannot align like the rigid short rods. However, the FFT image could confirm the orientation. It contributes to the optical anisotropy observed in C-POM. The electron diffraction result of the film can also prove the oriented structure. As shown in Fig. 2(c), from the width of the arc-like diffractions arising from the *ca.* 0.38 nm π-π stacking distance of the P3HT molecules, a mis-orientation of 20° of the fibril axis can be derived. P-UV-Vis absorption spectroscopy was also performed to calculate the orientation degree of the film quantitatively (Fig. 2d). It is noteworthy that two adjacent stripes were filled with the disordered P3HT nanofibrils. Therefore, the optical anisotropy could not be found in the zones between two adjacent stripes. As shown in Fig. 3, the diffraction ring is observed in the electron diffraction also indicating that the nanofibrils in the zone between two adjacent stripes are not oriented. In the FFT image, the orientation also could not be found.

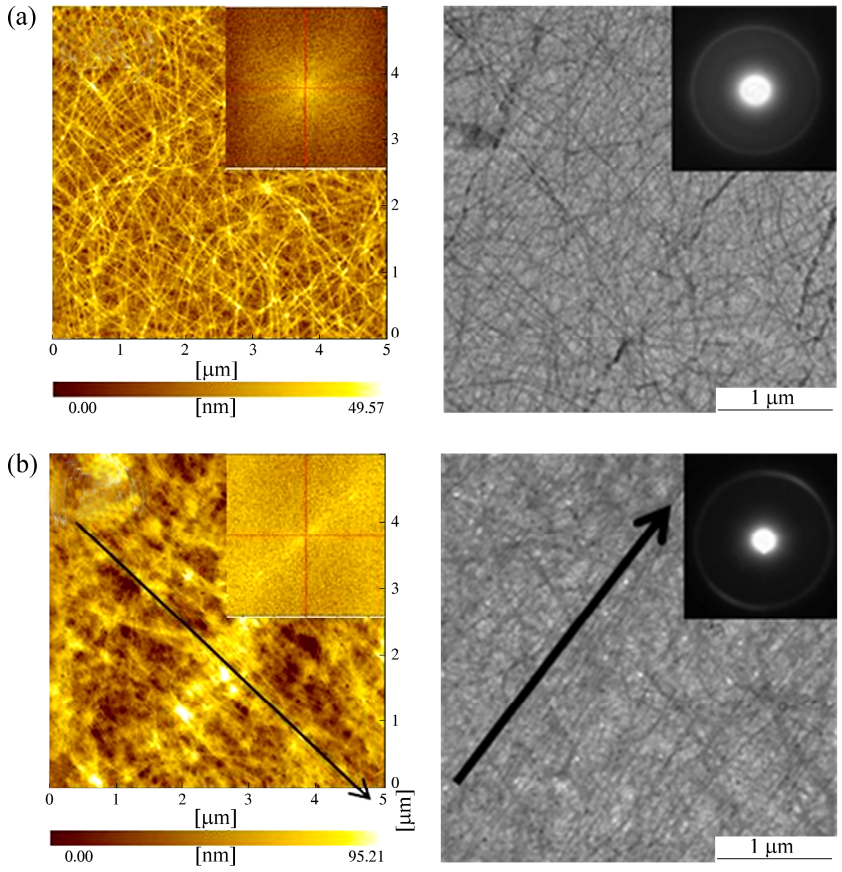


Fig. 3 AFM height images plus the corresponding FFT patterns (see inset) and TEM images plus the corresponding ED patterns (see insets) of the different zones of the film: (a) between the stripes and (b) on the stripe (The film was made by inclining glass substrate with an angle of 11.3°.)

As shown in Fig. 4, by using the hydrophilic substrate PEDOT, the oriented P3HT stripes could be gotten in the film like the film prepared with the glass substrate, it could be found that the P3HT nanofibrils in the stripe were perpendicular to the stripe. But when the hydrophobic substrate like OTS was used, the P3HT nanofibrils in the stripe were disorder. It also could be proved in the FFT image.

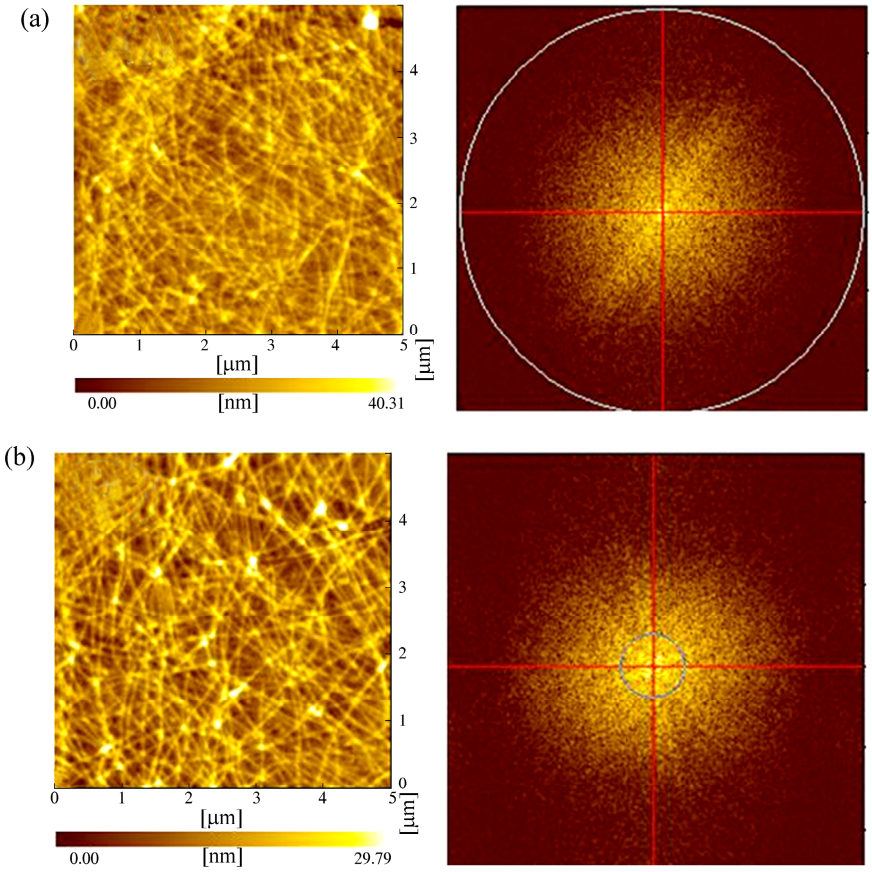


Fig. 4 The AFM height images and the corresponding FFT patterns on different substrates: (a) PEDOT substrate and (b) OTS substrate (The films were made by inclining substrate with an angle of 11.3°.)

The Orientation of Molecules in the P3HT Film

The P3HT molecules in the stripe structure take the edge-on orientation. In order to investigate the molecular orientation of the P3HT molecules in the films, all the three P3HT films were examined by using the GIXD. In the case of the pure P3HT sample, the intensities of the (100) reflection due to the lamellar layer structure and the (010) reflection due to π-π interchain stacking are strong, which indicates that the pure P3HT film contains a mixture of standing and lying polymer backbone chains[36]. Figure 5 shows the out-of-plane XRD patterns of the P3HT films. Here, the (100) reflection in three films was very strong, and the (010) reflection was negligible. We can conclude that the plane of crystalline P3HT is oriented preferentially normal to the substrate in the films (edge-on orientation). It seems that the P3HT molecules in the oriented P3HT films take the edge-on orientation both in the edge and the valley.

The SAED results can also prove the P3HT molecules take the edge-on orientation. As shown in Fig. 3, from the obtained SAED, we could only find the reflections arising from the *ca*. 0.38 nm π-π stacking distance of the P3HT molecules in the electron diffraction result of the film both in the edge and the valley. As is well known, these results corresponded to an ordered lamellar structure, which was formed by parallel stacks of P3HT main chains that were separated by the alkyl side chains. Meanwhile, we did not find the reflections arising from the *ca*. 1.56 nm alkyl-alkyl distance of the P3HT molecules. It means that the direction of the π-π interchain stacking was parallel to the substrate. Thus, we can deduce that part of the P3HT backbones adopted a parallel orientation with respect to the substrate, namely, edge-on conformation (no matter in the edge or in the valley).

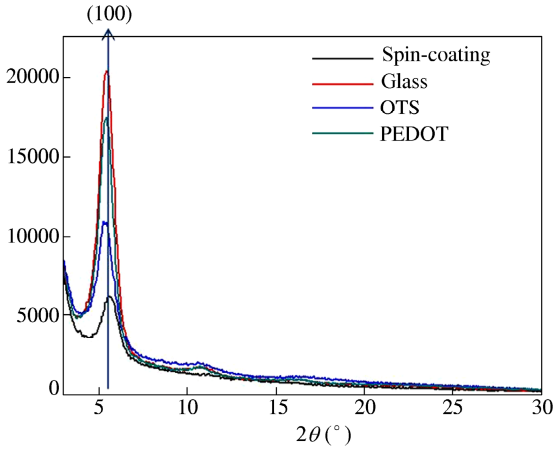


Fig. 5 Out-plane GIXD patterns of the films made by inclining glass, OTS, and PEDOT substrates and spin-coating method

DISCUSSIONS

The Formation of the Stripes Structure

The stripes structure could be obtained by controlled inclining evaporation. On the inclining substrate, the P3HT toluene solution drop was held by the surface tension with the highest evaporation rate at the edge of the drop. Only the three-phase contact line in the upper side can move because of the surface-tension and the gravity force. As the toluene evaporated, P3HT molecules were transported from the inside of solution to the edge of the drop and pinned at the contact line (*i.e.*, ''stick''), forming a stripe. During the deposition of P3HT, the initial contact angle gradually decreased to a critical contact angle, at which the surface tension (*i.e.*, depinning force) became larger than the pinning force[28], leading to the contact line to jerk (*i.e.*, ''slip'') to the next position inward, where it was pinned again and a new stripe was thus yielded. So, during the evaporation process, the solutes tend to segregate into concentrated, periodically distributed domains at the contact line.

The similar stripes structure could also form *via* droplet evaporation from a restricted geometry as shown in Scheme 1(b). With the evaporation of the droplet, the changing radius of curvature of the capillary bridge altered the instability condition between pinning and depinning of the contact line, the distance between adjacent stripes decreased fast. In our work, the drop was put on the inclining substrate and without the restricted geometry. The rate of the repetitive ''stick-slip'' motion of the three-phase contact line changed slowly during the toluene evaporation. The distance between adjacent stripes changed slowly before the concentration of the solution became too high and the solution changed to a gel.

With the increasing of the angle the speed, at which the initial contact angle gradually decreased to a critical contact angle, became fast because of the gravity. It makes the distance of the stripes become smaller with the increasing of the angle. When the angle was 5.7°, the distance was about 100 μm. When the angle was 11.3°, the distance was about 40 μm.

The Formation Mechanism of the Oriented Nanofibrils by Controlling the Wettability and the Inclining Angle

Because the P3HT lamellae diffuse directionally and then form the 1D crystal at the three-phase contact line of the drop, the oriented nanofibrils in the stripes can be obtained.

It has been pointed out that during the evaporation process the three-phase line recedes until it satisfies a receding contact angle for the solution on the solid substrate[37]. In our experiment, as the toluene evaporated, P3HT was transported from the inside of solution to the edge of the drop and pinned at the contact line. During the evaporation, the P3HT molecules separated from the solution would aggregate and crystallize first to form a nucleus in the edge of the three-phase contact line. With the suitable evaporation rate by controlling the inclining

angle and the wettability of the substrate, the peripheral P3HT lamellae would directionally diffuse owing to the directional evaporation of the solvent, which inhibited 2D growth. As a result, the P3HT crystal would adopt 1D growth and then form oriented nanofibrils. During the deposition of P3HT, the initial contact angle gradually decreased to a critical contact angle, at which the surface tension becomes larger than the pinning force, leading the contact line to jerk to the next position inward, where it was pinned again and a new stripe was thus yielded. During this process, a few P3HT solutions were residue between the two coffee rings. The residual P3HT solutions were evaporated without any limitation, the P3HT crystals would adopt 2D growth and then form the disordered nanofibrils.

The process of the directionally diffuse of P3HT lamellae and the formation of the 1D crystal was determined by the evaporation which is controlled by the wettability and the inclining angle.

The static contact angles (θ_s) of toluene on glass, OTS and PEDOT surfaces are smaller than 5° and 11.8° respectively. (Water spreads quickly on the glass. PEDOT and the OTS surface have a water contact angle of about 110°.) Though there is a large difference between the static contact angles, all of the P3HT films on these three substrates show the same structure of densely stacked nanofibrillar crystals.

The evaporation rate was decided by the contact angle of toluene to the substrate. There is a competitive relation between the evaporation and the crystallization in the meniscus zone. Only when the evaporation rate matches the 1D crystallization rate, the oriented P3HT stripes could be obtained by using the inclining substrate. In our experiment, the orientation of the P3HT stripes was found on the glass and the PEDOT surface but not on the OTS surface.

For forming the oriented P3HT nanofibrils, particularly, the solvent evaporation rate and crystallization rate must be chosen properly to obtain stationary conditions by changing the inclining angle. The shape of the drop will changed with increasing the inclining angle. The solvent evaporation rate in the meniscus region will become fast with increasing the inclining angle. With the small angle of inclination, the lower solvent evaporation rate in the meniscus region confined the orientation of the film, and the orientation of the nanofibrils could hardly be found in the AFM image and the low dichroic ratio D of 1.14 was obtained according to Eq. (1). But with the increasing of the angle, the solvent evaporation rate in the meniscus region became fast. With the faster solvent evaporation rate, the evaporation rate matches the crystallization rate, we can find the oriented nanofibrils in the AFM and TEM images. In the same time, we can find that the dichroic ratio D would increase to 1.89.

CONCLUSIONS

In summary, the P3HT stripe structure with oriented P3HT nanofibrils was prepared by a controlled inclining evaporative technique. The distance of the adjacent stripes could be controlled from 40 μm to 100 μm with decreasing the inclining angle. The degree of orientation could be attributed to two intrinsic parameters: the wettability of the substrate and the inclining angle. The hydrophilic substrate is beneficial to get the anisotropic P3HT stripe structure. The degree of orientation of the film was increased with the increasing of the angle. The highest dichroic ratio D could be gotten to 1.89, when the angle was 11.3°.

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