

Structure of Friction-transferred Highly Oriented Poly(3-hexylthiophene) Thin Films*

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Abstract Oriented thin films of P3HT were obtained by a friction-transfer technique. The morphology and structure of the film were studied by means of optical microscopy, atomic force microscopy and transmission electron microscopy. Optical microscopy observation indicates that large size well-ordered P3HT thin films can be produced by a friction-transfer technique. Highly ordered lamellae were observed in P3HT friction-transferred films by electron microscopy. Electron diffraction results confirm the existence of high orientation with the *a*- and *c*-axes of P3HT crystals aligned in the film plane while the *c*-axis parallel to the friction-transfer direction. The atomic force microscopy observation of the as-prepared P3HT thin film shows, however, a featureless top surface morphology, indicating the structure inhomogeneity of the obtained film. To get highly oriented P3HT thin films with homogenous structure, high temperature annealing, solvent vapor annealing and self-seeding recrystallization of the friction-transferred film were performed. It is confirmed that solvent vapor annealing and self-seeding recrystallization methods are efficient in improving the surface morphology and structure of the friction-transferred P3HT thin film. Highly oriented P3HT films with unique structure can be obtained through friction-transfer with subsequent solvent vapor annealing and self-seeding recrystallization.

Keywords: Poly(3-hexylthiophene); Friction-transfer; Thin film; Orientation.

INTRODUCTION

Regioregular poly(3-hexylthiophene) (P3HT) is one of the most popular organic semiconductors^[1–4]. Because of its special optical-electrical characteristics, P3HT can be used as the active layer in various thin film devices and sensors, *e.g.*, light-emitting diodes, organic solar cells, and field-effect transistors^[5–9]. Enormous efforts have been devoted to improve the charge transport or optical properties of P3HT thin films which were closely related to the morphology and particularly to the semicrystalline structure^[10–20]. The dependence of morphology and crystallinity of P3HT films on molecular weight (M_w) was considered as one of the most important issues for this correlation^[21–24]. Because the electrons are delocalized along the polymer backbone, conjugated polymer molecules in crystalline phase exhibit anisotropic properties^[25–30]. It is proved that the charge mobility of P3HT is higher along the polymer chain direction (*c*-axis of crystal unit cell) than in the direction of alkyl side-chains (*a*-axis)^[31]. The single crystals of P3HT with different spatial arrangements of backbone plane also show different charge transport properties^[32–34]. It is well documented that single crystals with well controlled unique structure are ideal for fabricating high efficiency optical-electrical devices. However, preparation of single crystals with sufficient size is hardly possible for polymers. In this case, from a practical standpoint, it is of great

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significance for a better modification of structural anisotropy for large area P3HT films, so as to fabricate high performance optical-electrical devices.

Two effective ways have been frequently reported to prepare oriented polymer films, *i.e.*, epitaxial overgrowth on oriented substrates^[35–39] and the friction-transfer technique^[40–42]. The P3HT can epitaxially crystallize on some substrates^[43–47]. As an example, highly oriented P3HT films have been successfully obtained through epitaxial crystallization on highly oriented PE films^[46]. It should be pointed out that the use of substrate thin films may affect the charge transport property of the devices. Therefore, friction-transfer technique has been used to produce highly oriented thin films of P3HT^[41]. The thus prepared thin P3HT films exhibit ideal orientation state with order parameter close to unity. It should be pointed out that the highly oriented friction-transferred P3HT film shows, however, featureless surface morphology under scanning electron microscope. Here in this work, friction-transfer oriented P3HT thin films were prepared and their surface and interior fine structures were studied by atomic force microscopy (AFM) and transmission electron microscopy (TEM). While the TEM clearly displays the edge-on lamellar structure of the ordered P3HT thin films, AFM observation shows also a featureless top surface morphology. This may indicate the existence of a thin amorphous layer of P3HT on the surface of the friction-transferred film, which can influence the performance of the thin film. It was well known that annealing and self-seeding were widely used methods to modify the morphology and structure of polymers^[48–51]. To improve the top surface structure of the friction-transferred P3HT thin films, the annealing at elevated temperatures, self-seeding recrystallization and solvent vapor annealing methods were also utilized in this work. Through these processes with controlled crystallization or annealing conditions, parallel aligned edge-on lamellar structure both at the surface and interior of the film has been obtained.

EXPERIMENTAL

Materials

P3HT with high molecular weight ($M_w = 87$ kDa) was purchased from Sigma-Aldrich, and the head-to-tail regiospecific conformation is more than 99%. All solvents used here were purchased from Beijing Chemical Works.

Preparation

The original P3HT sample was dissolved in chloroform with a concentration of 1% (*W/V*) and a thick film was obtained after the evaporation of the solvent. The friction-transfer process was carried out by squeezing and drawing the P3HT thick film manually with a stick on a glass slide at 200 °C. The surface morphology modification of the friction-transferred P3HT thin films has been conducted in three different ways: 1) annealing at elevated temperatures; 2) recrystallization through self-seeding; and 3) annealing in different solvent vapor environments. For the self-seeding recrystallization, the films were first heated to 230 or 250 °C for 3 min and then cooled down to 130, 150 or 170 °C at a rate of 40 K/min for 30 min isothermal recrystallization. For solvent vapor annealing, the films were annealed at 40 °C for 24 h in solvent vapor atmosphere of acetonitrile, *p*-xylene and tetrahydrofuran (THF), respectively.

Characterization

The optical microscopy images were obtained by using an Axioskop 40A Pol optical microscope (Carl Zeiss) under crossed polarizers. The surface morphology of P3HT films was studied by an Agilent Technologies 5500 atomic force microscope (Agilent Technologies Co. Ltd., U.S.) at room temperature in air. The images were obtained by means of tapping mode with a silicon cantilever having a spring constant of 20–30 N/m and a resonating frequency of 320–350 kHz, and the scanning rates varied from 2 $\mu\text{m/s}$ to 5 $\mu\text{m/s}$. For transmission electron microscopy (TEM) examination, the friction-transferred film was detached from the substrate with the help of poly (acrylic acid) (PAA) and mounted onto 300 mesh TEM copper grids. TEM experiments were performed using a JEOL JEM-2100 electron microscope operated under an accelerating voltage of 200 kV.

RESULTS AND DISCUSSION

Morphology of the As-prepared P3HT Friction-transferred Films

The morphology of friction-transferred P3HT films was first studied by optical microscopy. Figure 1 provides the polarized optical micrographs of an as-prepared friction-transferred P3HT thin film with the sliding directions inclined at angles of 45° and 0° to the polarization direction, respectively. As we can see from Fig. 1(a), when the sliding direction is oriented at 45° to the polarizer, the film exhibits very strong birefringence. However, complete extinction happens when the sample is rotated about the light beam for 45° , leading to a parallel alignment of the sliding direction along the polarization direction. According to the role of light extinction under polarized optical microscope, this results indicate a perfect orientation of the P3HT molecules in the friction-transferred film. Moreover, the optical micrographs of the friction-transferred P3HT film have a relatively smooth surface. This indicates the feasibility of friction-transfer technique for producing large size well-ordered P3HT thin films with exceptional anisotropic optical property, which can reach square centimeter in size.

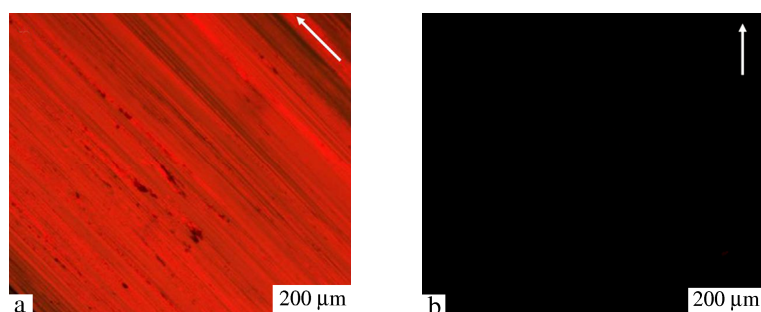


Fig. 1 Polarized optical micrographs of a friction-transferred P3HT film with the rubbing directions (indicated by the arrows) inclined (a) 45° and (b) 0° to the polarization direction, respectively

To view the fine structure of the friction-transferred P3HT thin film, AFM observation was conducted. Figure 2(a) shows a top surface AFM height image of a friction-transferred P3HT thin film. The AFM height image shows a uniform top surface morphology. It is featureless, which has a close resemblance with the SEM image reported by Nagamatsu and coworkers^[41]. To reveal its bottom surface morphology, the friction-transferred P3HT thin film was detached from the glass slide with the help of PAA and monitored also by AFM. As presented in Fig. 2(b), the bottom surface of the film shows a clearly different morphology as compared with the top surface. It consists of lamellar structure with short lamellae aligned perpendicular to the sliding direction, indicating an orientation of molecular chains along the sliding direction.

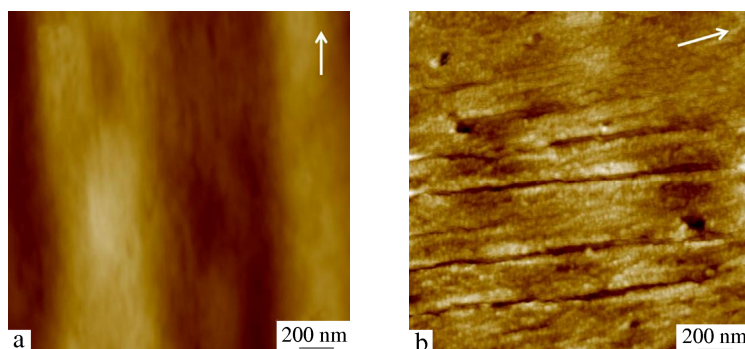


Fig. 2 AFM height images of the (a) top and (b) bottom surface morphologies of an as-prepared friction-transferred P3HT film

The fine structure of the as-prepared P3HT friction-transferred film was also studied by TEM combined with electron diffraction. Figure 3(a) shows a representative bright field (BF) phase contrast electron micrograph of a P3HT friction-transferred film. It consists of oriented lamellae (gray lines) aligned perpendicular to the sliding direction (indicated by the white arrow), indicating a high orientation of the P3HT friction-transferred film. This implies that sliding at elevated temperatures is in favor of the formation of oriented lamellar structure of P3HT. The corresponding electron diffraction pattern, Fig. 3(b), shows well defined reflection spots. This further confirms the existence of high orientation of the prepared friction-transferred P3HT film. All of the diffraction spots are accounted for by an orthorhombic unit cell with lattice constants ($a = 1.663$ nm, $b = 0.775$ nm, $c = 0.777$ nm, $\alpha = \beta = \gamma = 90^\circ$)^[52]. The location of the (002) reflections in the sliding direction indicates that the molecular chains of P3HT lie in the film plane and along the sliding direction. The appearance of strong (100), (200) and (300) reflections further indicates that the a -axis of P3HT crystals in the friction-transferred film is oriented in film plane. This means that the as-prepared P3HT film exhibits a double orientation with crystallographic b -axis perpendicular to the film plane^[39, 53]. A schematic illustration of the P3HT chain arrangement in the friction-transferred film is presented in Fig. 3(c). It is different from the films self-assembled from solution, where an a -axis orientation is identified. This should be attributed to the strong shearing force exerted in the sliding process. The strong shearing force encourages the thiophene rings in P3HT molecular chain lying down to the substrate. Moreover, from the electron diffraction pattern, the distributions of the (002) and (h 00) reflections of the friction-transferred P3HT film were estimated to be about 10° , which demonstrate the excellent orientation of the film.

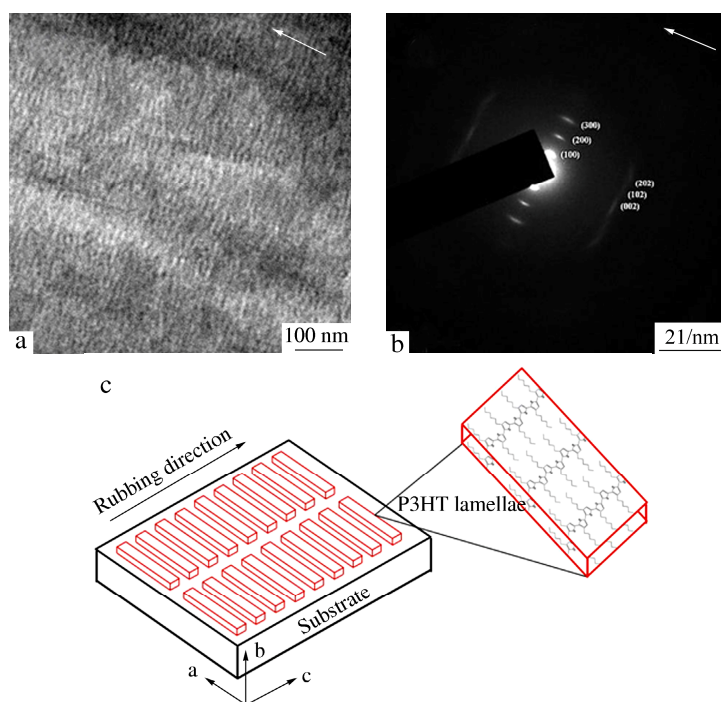


Fig. 3 (a) A BF electron micrograph and (b) its corresponding electron diffraction pattern of an as-prepared P3HT film (The sliding directions were indicated by white arrows.); (c) Schematic illustration of the P3HT chain arrangement in the friction-transferred film

From the above electron microscopy results, it is clear that the top surface structure of the friction-transferred P3HT film is different from its inside and bottom surface structure. This may imply the existence of a thin amorphous top surface layer. The existence of an amorphous surface layer may influence the charge transport properties. To get fully oriented structure, further treatments were performed for the friction-transferred films.

Morphology of Friction-transferred P3HT Films Annealed at Different Temperatures

As discussed above, the top surface morphology of the friction-transferred P3HT film is different from that of the inside film and bottom layer. Therefore, efforts have been made to improve the structure of the top surface layer. It was well documented that annealing at elevated temperatures can realize structure rearrangement of polymeric materials, which may promote the recrystallization of the top surface layer of the friction-transferred P3HT thin films. Therefore, the friction-transferred P3HT films were annealed at 130 °C, 150 °C and 170 °C, respectively. Figure 4 shows the AFM phase images and TEM BF images of the friction-transferred P3HT films annealed at different temperatures for 3 h. From the BF electron micrographs, we can see that annealing at 130 °C and 150 °C for 3 h does not change the morphology of the friction-transferred P3HT thin film very much. The sample annealed at 170 °C for 3 h exhibits better arranged lamellae with somewhat stronger contrast. This may be related to an improved crystalline structure of P3HT. On the other hand, the AFM images show some tiny changes of the top surface. It seems that there exist microcrystallites on the surface of the friction-transferred P3HT thin films, indicating the rearrangement of the surface layer. Nevertheless, oriented lamellar structure is still absent. This means that the structure reorganization is not quite effective by annealing at elevated temperatures.

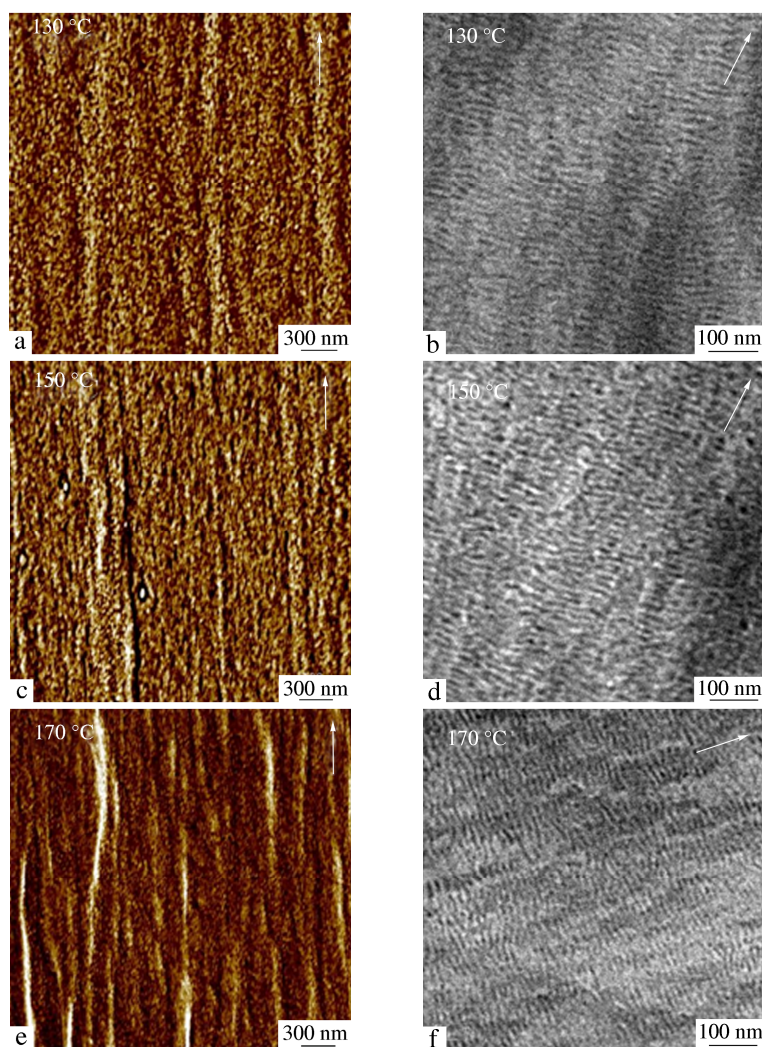


Fig. 4 AFM phase images (left column) and TEM BF images (right column) of P3HT friction-transferred thin films after annealing for 3 h at temperatures of 130 °C (top), 150 °C (middle) and 170 °C (bottom) (The arrows indicate the molecular chain directions of P3HT in the films.)

Morphology of Friction-transferred P3HT Films Treated through Self-seeding

Self-seeding provides an efficient way for producing well organized structures of polymer thin films. Reiter *et al.* have successfully cloned the crystal structures of P2VP-*b*-PEO and PFS by self-seeding crystallization^[54]. In order to fabricate high oriented P3HT thin films with unique structure through a friction-transfer technique, self-seeding was employed for recrystallizing the friction-transferred P3HT thin films. As well known, the morphology and structure of crystals depend only on the crystallization conditions but have nothing to do with the former crystals if they are melted completely. However, in the self-seeding procedure, the crystals are not melted completely. In this case, some self-nuclei of the previous crystals are preserved. These self-nuclei are able to make the subsequently grown crystals have a similar morphology and structure with the original ones when crystallization later ensues. In this way, highly oriented P3HT thin films with uniform oriented structure are expected after self-seeding recrystallization.

Figure 5 shows an AFM height image and the UV-Vis absorption spectra of a friction-transferred P3HT thin film, which was melted at 250 °C for 3 min and subsequently recrystallized at 150 °C for 30 min. The AFM image reveals the lamellar structure of the top surface, indicating the occurrence of recrystallization. However, the random orientation of the lamellae indicates that the thermal treatment has dispelled the ordered structures. In the UV-Vis absorption spectra (see Fig. 5b), the blue shift of the melt recrystallized film also indicates the elimination of orientation. This implies that the friction-transferred P3HT thin film is completely melted at 250 °C. It is acceptable since the chosen temperature is about 10 K above the melting point of P3HT (~240 °C).

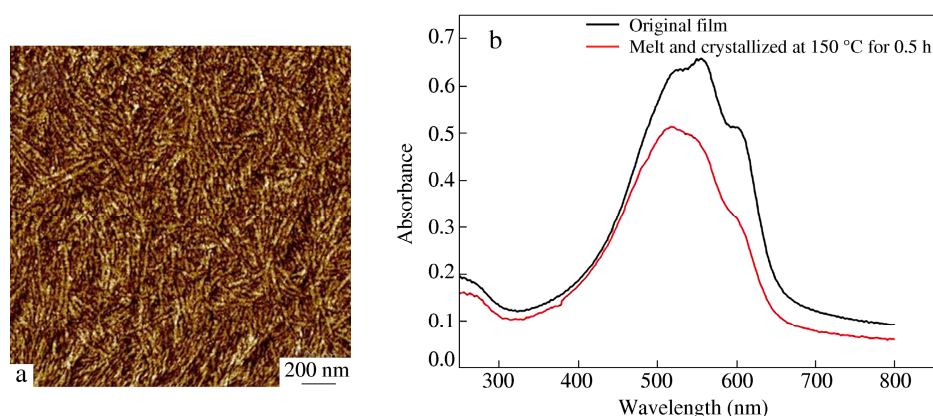


Fig. 5 P3HT friction-transferred film melted at 250 °C for 3 min and recrystallized at 150 °C for 30 min: (a) AFM height image; (b) UV-Vis absorption spectra

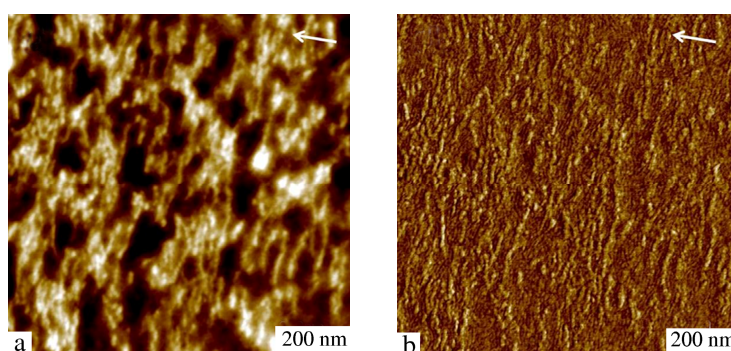


Fig. 6 AFM (a) height and (b) phase images of a friction-transferred P3HT thin film melted at 230 °C for 3 min and then recrystallized at 150 °C for 30 min (The white arrows in the images indicate the sliding direction during sample preparation.)

Nevertheless, it helps to distinguish the self-seeding recrystallization process presented hereafter from the melt recrystallization behavior. Figure 6 shows the AFM height and phase images of a friction-transferred P3HT thin film, which melted at 230 °C for 3 min and then recrystallized at 150 °C for 30 min. Now, long and well oriented lamellae of P3HT were observed, indicating the propagation of oriented lamellar structure from underneath layer to the top surface layer. This demonstrates the success of self-seeding technique in regulating the structure of the friction-transferred P3HT thin films.

Morphology of Friction-transferred P3HT Films after Solvent Vapor Annealing

Another widely used method for promoting the crystallization of polymers is known as solvent vapor annealing. In this process, the solvent vapor will diffuse to some extent into the surface layer and enhance chain mobility of the polymer in surface layer, which is benefit for the reorganization of molecular chains in amorphous phase into crystalline state. Actually, the solvent vapor annealing has already been used to grow needle-like P3HT single crystals^[34, 55]. Its advantage is that the molecular chains on the top layer of the friction-transferred film will be sufficiently affected by the solvent vapor, and the inner oriented structure of the film will hardly be destroyed. In the present work, acetonitrile, *p*-xylene and THF were chosen as the solvents. The friction-transferred P3HT thin films were put into a container with a vapor environment of the chosen solvent at 40 °C for 24 h. Figure 7 shows the AFM phase images of the friction-transferred P3HT thin films treated with different solvent vapors as indicated. Comparing with the as-prepared samples, some changes of the surface morphology of P3HT thin films can be identified after the treatment. For the acetonitrile vapor treated sample, some microcrystallites of P3HT on the surface of the friction-transferred film can be recognized. The *p*-xylene vapor treated thin film exhibits a similar surface morphology as the acetonitrile vapor treated one with plenty of microcrystallites. In the AFM image of the THF vapor treated friction-transferred P3HT thin film, lamellar crystals can be observed. The lamellae have a close resemblance with those observed by TEM, indicating higher extent of structure rearrangement. These P3HT lamellae are mostly oriented in the direction perpendicular to the sliding direction. In some regions, the P3HT lamellae are not well oriented. This may be caused by the inhomogeneity of the friction-transferred films. There may be poorly crystallized thinner regions, where the molecular chains are fully relaxed during solvent vapor treatment. In this case, recrystallization will result in a random orientation of the crystalline lamellae. The complete relaxation of molecular chains may also happen in the top layer of the thicker films. But the underneath layer keeps its high orientation. The intact underneath layer of friction-transferred P3HT film will serve as an oriented substrate and initiate homoepitaxial crystallization of the relaxed P3HT molecules. This causes the lamellae grow in the same orientation as the friction-transferred film. The above results indicate that the solvent vapor is helpful for chain reorganization and promotes the crystallization of P3HT in the top surface layer. The difference in the reorganized surface morphologies should originate from the different solubility of the solvent toward P3HT. The solubility of acetonitrile and *p*-xylene are poor for P3HT.

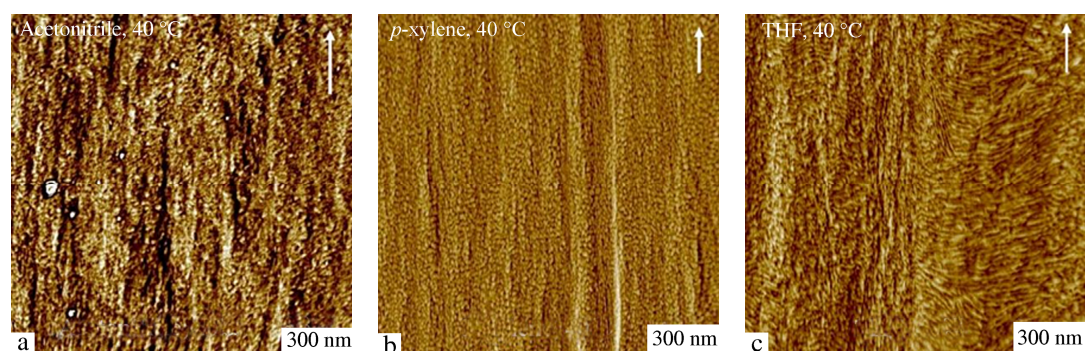


Fig. 7 AFM phase images of friction-transferred P3HT films after annealing at 40 °C for 24 h in different solvent vapors as indicated (The white arrows in the images indicate the friction-transfer direction during sample preparation.)

Their effects on improving the chain mobility of P3HT are limited. As a result, only microsize crystalline grains have been produced after the solvent vapor annealing treatment. On the contrary, the THF has a higher affinity to P3HT. Therefore, its vapor may encourage a better rearrangement of the P3HT molecules in the top layer, leading to the formation of larger size crystalline lamellae. The electron diffraction result also demonstrates an improved orientation of the friction-transferred P3HT film treated under THF atmosphere. As presented in Fig. 8, the diffraction spots become sharper and clearer than those appeared in the pattern of as-prepared friction-transferred P3HT film (Fig. 3b). Now the distributions of the (002) and (*h*00) reflections of the friction-transferred P3HT film decreased to about 9°, indicating an increased orientation of the P3HT film.

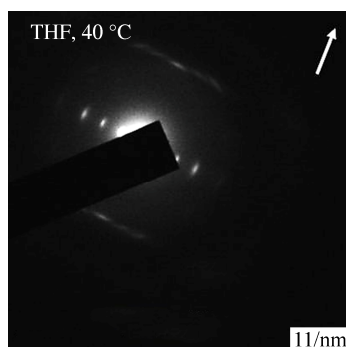


Fig. 8 An electron diffraction pattern of a friction-transferred P3HT film after annealing in THF solvent vapor at 40 °C for 24 h (The white arrow in the image indicates the sliding direction during sample preparation.)

CONCLUSIONS

Highly oriented P3HT films were made by a friction-transfer technique at elevated temperatures. Electron microscopy observation shows that well aligned lamellae of the friction-transferred P3HT thin film, indicating the high orientation of the obtained films. The diffraction pattern displays well defined reflection spots and confirms the existence of high orientation. Indexing of the diffraction spots demonstrates that the *a*- and *c*-axes of the P3HT crystals lie in the film plane with the *c*-axis oriented in the sliding direction. The light extinction phenomenon of the samples under optical microscope with crossed polarizers when the sliding direction aligned in different directions related to the polarizer also suggests a perfect orientation of the obtained friction-transferred P3HT thin films. The relatively smooth surface topology of the optical micrographs tells us that large area highly oriented P3HT thin film can be fabricated through a friction-transfer technique. The AFM results show, however, different top and bottom surface structures of the friction-transferred P3HT thin films. While oriented lamellar structure similar to that shown in the electron micrograph was observed on the bottom surface, no crystalline morphology was identified on the top surface. This indicates the structure inhomogeneity of the friction-transferred P3HT thin film. The reason for the formation of different top surface structures is not clear at the moment. It has, however, been successfully modified through self-seeding and solvent vapor annealing treatments. Through self-seeding recrystallization, oriented lamellar structure in the top surface layer of the friction-transferred P3HT thin film has been created. Also the THF solvent vapor annealing is in favor of the structure reorganization of the P3HT in top surface layer and encourages the formation of long oriented lamellae. This may be related to the homoepitaxial crystallization of the top surface layer on the highly oriented underneath layer produced by friction-transfer.

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