

Effect of Solvent Annealing Temperature on Crystal Modifications and Phase Transition Behavior of Regioregular Poly(3-octylthiophene)^{*}

Xiao-jun Wang, Yuan Yuan, Li-li Han, Xia Dong and Jian-ming Zhang^{**}

Key Laboratory of Rubber-Plastics, Ministry of Education/Shandong Provincial Key Laboratory of Rubber-plastics,
Qingdao University of Science & Technology, Qingdao 266042, China

Abstract In present study, the effect of the solvent annealing temperature on the crystal modifications and the phase transition behavior of the subsequently dried poly(3-octylthiophene) (P3OT) film has been investigated by the combination of DSC, WAXD and FTIR techniques. When chloroform is employed as the solvent, it is unexpectedly found that form I and form II crystal modifications of P3OT could be respectively obtained by room temperature and low temperature annealing. Comparing to the mostly used solvent reported for preparing form II, *i.e.* carbon disulfide (CS₂) which is toxic and corrosive, chloroform is less toxic and corrosive and more suitable for solution processing of P3OT. Therefore, this finding provides an alternative way to obtain form II. By temperature dependent IR spectroscopy, the structural evolution of P3OT during the form II to form I phase transition process has also been studied in detail.

Keywords: P3OT; Crystal modification; FTIR.

INTRODUCTION

Regio-regular poly(3-alkylthiophene)s (rr-P3ATs) have been used extensively in organic electronic applications as active layers because of their excellent solution processability and high field-effect mobility^[1, 2]. As a kind of crystalline polymers, the crystalline structure and morphology of P3ATs significantly affect the thin film charge transport properties and consequently dominate the device performance. Compared to the linear polymers, the comb-like P3ATs own complex hierarchical structures including main chains acting as the stiff backbones and flexible alkyl side chains endowing them with high solubility in common organic solvents^[3]. Previous studies show that the crystalline structure of P3ATs could be influenced by many factors, such as regioregularity^[4], molecular weight^[5], and thermal or solvent treatment^[6].

In general, P3ATs can form three kinds of crystal structures, named as form I, form I' and form II, which differ mainly in interlayer distance along the a-axis. Form I is the most common crystal modification of P3AT and could be easily obtained by solution or melt-crystallization whereas the formation of form I' and form II requires specific sampling conditions, which have been reported by several groups. For example, Prosa *et al.*^[7] first found that form II of poly(3-octylthiophene) (P3OT) and poly(3-dodecylthiophene) (P3DDT) could be obtained by casting film from 1%–3% (*W/W*) solution in xylene at room temperature. Subsequently, Yang *et al.*^[8] reported that low evaporation rate from CS₂ leads to formation of poly(3-butylthiophene) (P3BT) form II crystals with spherulitic morphology. Recently, they also demonstrated that by solution aging, the P3BT

^{*} This work was financially supported by the Taishan Mountain Scholar Constructive Engineering Foundation (No. TS20081120) and Natural Science Fund for Distinguished Young Scholars of Shandong Province (No. JQ200905).

^{**} Corresponding author: Jian-ming Zhang (张健明), E-mail: zjm@qust.edu.cn

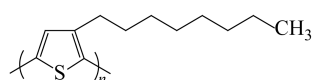
Invited paper for the special issue of “Polymer Crystallization”

Received March 13, 2014; Revised April 22, 2014; Accepted April 28, 2014

doi: 10.1007/s10118-014-1493-y

molecules arrange into form II structure in high boiling solvents, such as *o*-dichlorobenzene (ODCB), *p*-xylene and CB^[9]. We recently also reported that the form I' of P3BT and P3HT can be obtained by at slow solvent evaporation rate and/or suitable annealing temperature^[10, 11]. These studies clearly show that solvent annealing treatment is very useful for controlling the crystal structure of P3ATs.

In the present study, we investigate the effect of the solvent annealing temperature on the crystal modifications of P3OT, in which there is a long alkyl side chain as shown in Scheme 1. Considering that some organic solvents, such as CS₂, ODCB *etc.* are extremely toxic and corrosive, chloroform is employed as the annealing solvent. To one's surprise, it was found that form II can be obtained at relatively low annealing temperatures. This finding was confirmed by DSC, WAXD and FTIR techniques. Furthermore, the structural evolution and the phase transition behavior of P3OT form II were also studied in detail by temperature-dependent IR spectroscopy. This study provides new strategies for controlling polymorphs of P3OT using solvent annealing and subsequent thermal annealing.

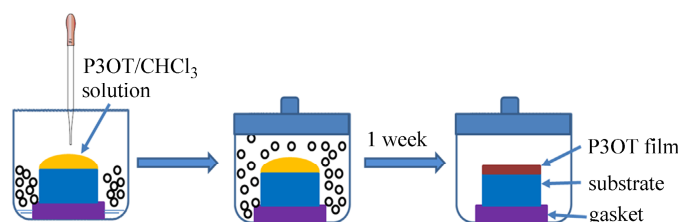


Scheme 1 Molecular structure of P3OT

EXPERIMENTAL SECTION

Material and Sample Preparation

Regio-regular poly(3-octylthiophene) (ADS308PT, $M_w = 35000$ g/mol and $M_w/M_n = 3.8$, regioregularity is 95%–98%) was purchased from American Dye Source, inc. P3OT was first added in chloroform (concentration 40 mg/mL) and stirred on a hot stage at 40 °C until completely dissolved. The P3OT film was prepared by casting several drops of solutions uniformly overspreading the substrate kept inside a cylinder container with a radius and height of 1.5 and 3 cm, respectively. Meanwhile, 0.3 mL chloroform was deposited around the substrate to generate chloroform partial pressure. Subsequently, the containers with samples were sealed and kept at different temperatures, *i.e.* room temperature (25 °C), 10 °C and –5 °C. The sampling method is illustrated in Scheme 2. Generally, it takes about one week for the majority of solvent evaporated and the P3OT films turned to dry, then the samples were placed under vacuum at room temperature for 24 h to remove residual solvent completely.



Scheme 2 Illustrations of sampling process for the solvent annealing P3OT films

Characterizations

DSC measurements were performed on a TA Q20 calorimeter system over a temperature range from –50 °C to 250 °C under flowing nitrogen gas at a heating rate of 10 K/min.

Wide-angle X-ray diffraction (WAXD) was performed using the Rigaku SmartLab X-Ray Diffractometer with Cu K α radiation ($\lambda = 0.154$ nm). WAXD profiles were taken in the 2θ range from 2° to 30° by continuous scanning with a step size of 0.05°.

FTIR spectra were measured with a Bruker tensor 27 spectrometer equipped with a DTGs detector. The normal transmission mode was employed. For studying the thermal behavior of P3OT *via in situ* FTIR, the as-prepared film on KBr substrate was set on a Harrick variable temperature cell, which was placed into sample

compartment of the spectrometer. Subsequently, the sample was heated at a rate of 2 K/min from room temperature to 210 °C in nitrogen atmosphere. During the heating process, FTIR spectra of the specimen were recorded at a 1 min interval from 30 °C to 210 °C. The spectra were obtained by coadding 16 scans at a 2 cm⁻¹ resolution.

The intensities of IR bands were calculated automatically by a numerical data processor program for vibration spectroscopy, Spina Version 3, which was developed by Yukiteru Katsumoto in the Ozaki Group of Kwansei-Gakuin University. Before the intensity calculation, all of the spectra were baseline corrected. The difference spectra and second derivatives curves were performed by the IR measurement and data processor program- Bruker OPUS Version 7.2.

RESULTS AND DISCUSSION

Effect of Solvent Annealing Temperature on Crystal Modification Investigated by WAXD, PLM and FTIR

It is well-known that the crystal modifications or crystal structure of P3ATs could be affected by these various sampling procedures and the subsequent thermal annealing process, in which the solvent evaporation rate and the solvent annealing temperature play an important role. Since slow evaporation rates will improve the structure ordering or enhance the crystallinity, we mainly investigated the effect of solvent annealing temperature on the crystal modification at slow evaporation rates and the sampling procedure is described in experimental section. Figure 1 displays the WAXD profiles of P3OT films obtained at 25 °C (room temperature), 10 °C and -5 °C respectively. It can be seen that the characteristic (100) diffraction peak of form I modification located at $2\theta = 4.4^\circ$ is observed for the P3OT film annealed by chloroform at 25 °C^[12]. However, for P3OT films annealed at 10 °C and -5 °C, these form I diffractions are absent, and a significant diffraction peak is located at $2\theta = 6.1^\circ$, which confirms that form II is the dominant crystal modification^[7]. Generally, the main difference between these two crystal modifications is the shorter a-axis repeat distance of the form II crystals ($d_{100} = 1.45$ nm) as compared with that of form I crystals ($d_{100} = 1.96$ nm) resulting from interdigitation or tilting of the side chains. It has been reported that the crystallographic transitions of P3ATs from form I to form II take place as the specimen is exposed to CS₂ vapor for a long time^[13, 14]. Meanwhile, almost all the form II P3ATs have been formed at room temperature. Surprisingly, in our experiment, when chloroform replaces CS₂ as the annealing solvent, form I modification rather than form II is obtained at room temperature and form II is only discovered at relatively low temperatures. In general, the far more commonly obtained form I P3AT crystal is the thermodynamically stable form compared with form II. On the one hand, we speculate the crystal modification of P3OT depends on the solvent annealing temperature, which could be relevant to the

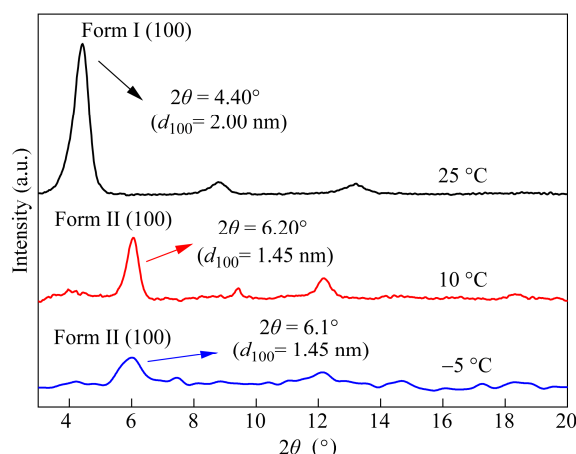


Fig. 1 WAXD profiles of the P3OT films annealed by chloroform at 25 °C, 10 °C and -5 °C, respectively

thermodynamic stability of the various forms. That is, the low temperature does not favor the movement of molecules and thereby inhibits the formation of more stable form I. On the other hand, the effect of solvent on the crystal modification could be related to the solubility of the solvent. However, the detailed reasons have not been identified clearly and need to be studied further. Therefore, we can conclude that when chloroform is used as the annealing solvent, form I and form II modification of P3OT can be obtained at room temperature and lower temperatures, respectively.

It has been widely accepted that the form II P3ATs often grow into spherulitic morphology, so we perform polarized light microscopy observation to investigate the morphology of the form II P3OT film prepared at 10 °C as shown in Fig. 2. It is obvious that spherulites with diameter of about 10 μm and the typical Maltese cross pattern can be clearly seen. It suggests that upon low temperature annealing (10 °C), the slow solvent evaporation rate allows generation of a few nuclei which gradually grow into spherulites. This result is in line with the previously reported studies that the P3BT form II films appear in the typical spherulitic morphology^[9, 13]. The size of spherulites is dependent on the sampling procedures and post annealing conditions.

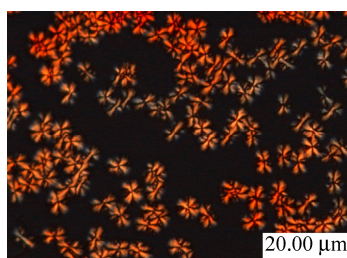


Fig. 2 The polarized light microscopy image of a P3OT film annealed by chloroform at 10 °C

More evidence to confirm the existence of different P3OT modifications after annealing at different temperatures can be revealed by infrared spectroscopy, which is sensitive to local molecular structures in semicrystalline polymeric systems. The FTIR spectra of the P3OT films annealed by chloroform at 25 °C, 10 °C and −5 °C are displayed in Fig. 3. Obviously, there are three spectral regions: 3000–2800 cm^{-1} , C–H stretching modes of alkyl side chain; 1550–1400 cm^{-1} , the C=C stretching vibration modes of conjugated backbones; 850–800 cm^{-1} , the out-of-plane deformation modes of thiophene $\text{C}_\beta\text{—H}$ sensitive to the crystal structure of P3ATs^[10, 15]. The three spectra are almost identical, except in the $\text{C}_\beta\text{—H}$ out-of-plane bending region, highlighted in the figure inset. The frequency at the peak maximum is 826 cm^{-1} for P3OT films prepared at 10 °C and −5 °C compared with 820 cm^{-1} for P3OT films prepared at 25 °C. Notably, these two bands have been assigned to the form II and form I crystalline structures of P3OT, respectively^[10, 11]. Therefore, the results further confirm our previous conclusions analyzed from WAXD data.

Effect of Solvent Annealing Temperature on Thermal Behavior of P3OT Films

Considering that the P3OT films with various crystal modifications should demonstrate different thermal stability, DSC analysis was subsequently conducted to study the thermal behavior of P3OT films annealed by CHCl_3 at different temperatures. At first, we take a close look at the DSC heating curves shown in Fig. 4. Remarkably, there are two endothermic transitions in the DSC heating process for form II P3OT crystals prepared at 10 °C and −5 °C, different from the one endothermic transition of form I P3OT crystals prepared at 25 °C. At present, the similar phenomenon of double endothermic transitions has been reported for P3ATs. For instance, Su *et al.* found that the origin of two endothermic transitions for P3DDT depends on the molecular weight and crystal modifications. For form II P3DDT with high molecular weight (60000 g/mol), the endothermic transition at low temperatures is assigned to the conversion of form II to form I^[16]. But for form I P3DDT films with high molecular weight (60000 g/mol or 15000 g/mol), the endothermic transition is associated with a side chain melting^[17]. Herein, it can be reduced that for the form II P3OT crystal ($M_w = 35000$ g/mol) with shorter side chain, the first endothermic peak at 50–100 °C may be attributed to a phase

transition which will be given more evidence in the FTIR data analysis and the other peak located around 190 °C is related to the melting of polymer.

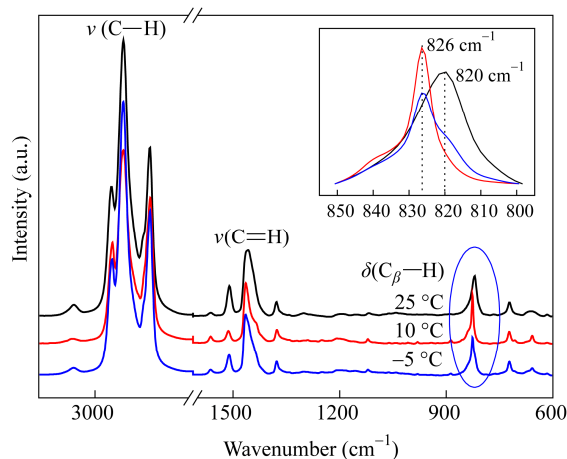


Fig. 3 IR spectra of the P3OT films annealed by chloroform at 25 °C, 10 °C and -5 °C in the region of 3000–600 cm^{-1} (The inset graph shows the local spectra in the 850–800 cm^{-1} region.)

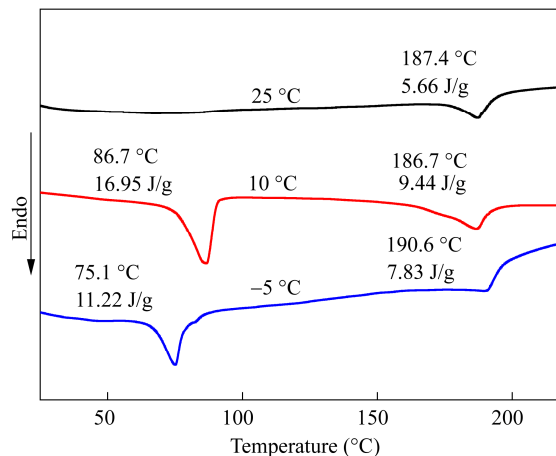


Fig. 4 DSC first heating curves of the P3OT films annealed at 25 °C, 10 °C and -5 °C, respectively

Combing with the WAXD, FTIR and DSC data, we can make the conclusion that form I and form II of the P3OT films can be respectively obtained at room temperature and relatively low annealing temperatures when the available solvent-chloroform is used. However, to the best of our knowledge, the endothermic behavior of form II P3OT in the low-temperature region has not been investigated in detail yet. Therefore, for clarifying the concrete structure changes corresponding to this endothermic transition at low temperatures, the temperature-dependent IR measurements were performed as follows.

Structural Changes of the Form II to Form I Phase Transition Investigated by Temperature-dependent FTIR Spectroscopy

FTIR is an efficient method to investigate the microstructures and phase transitions of polymers at the molecular level. Recently, it has been shown that this technique offers the unique advantage of exploring the structure of the main chain and side chain, and studying the crystalline and amorphous phases of the P3ATs^[18–20]. Meanwhile, according to references and our observation^[19, 21, 22], most of the IR characteristic bands of P3OT are listed in Table 1. Thus temperature-dependent FTIR spectroscopy is employed to explore the origin of the endothermic behavior around 87 °C and the corresponding structural changes upon heating for the solvent annealing P3OT film prepared at 10 °C.

Table 1. Bands assignment related to the structural transition for P3OT	
IR frequency (cm^{-1})	Assignment
2955	$\nu_{\text{as}}(\text{CH}_3)$
2870	$\nu_{\text{s}}(\text{CH}_3)$
2855	$\nu_{\text{s}}(\text{CH}_2)$
1510	$\nu_{\text{as}}(\text{C}=\text{C})$
1456	$\nu_{\text{s}}(\text{C}=\text{C})$
836	$\delta(\text{C}_\beta-\text{H})$ in amorphous phase
826	$\delta(\text{C}_\beta-\text{H})$ in crystalline phase-form II
820	$\delta(\text{C}_\beta-\text{H})$ in crystalline phase-form I

Structural change associated with octyl side chains

Figure 5 shows the spectral changes of the C—H stretching vibration mode in the region of 3000–2800 cm^{-1} in the heating process from 50 $^{\circ}\text{C}$ to 90 $^{\circ}\text{C}$ for P3OT form II. In this region, the CH_3 symmetric ($\nu_s(\text{CH}_3)$) stretchings and the CH_2 symmetric ($\nu_s(\text{CH}_2)$) stretchings located at 2870 cm^{-1} and 2855 cm^{-1} respectively, are characteristic of the alkyl side chains. As seen from the original spectra at the top of Fig. 5(a), there is a subtle peak shift toward higher frequencies for the $\nu_s(\text{CH}_3)$ band but no obvious peak shift for the $\nu_s(\text{CH}_2)$ band. For clearly comparison, the corresponding difference spectra by the subtraction of the initial spectrum measured at 50 $^{\circ}\text{C}$ is depicted at the bottom of Fig. 5(a). Obviously, the appearance of a positive peak located at 2876 cm^{-1} and a negative peak located at 2867 cm^{-1} in the difference spectra contributes to the apparent peak shift of the $\nu_s(\text{CH}_3)$ band around 2870 cm^{-1} in the original spectra. Meanwhile, as plotted in Fig. 5(b), the intensity of the peak located at 2876 cm^{-1} calculated from the original spectra increases, whereas the intensity of the peak at 2867 cm^{-1} decreases simultaneously in the temperature region of 50 $^{\circ}\text{C}$ to 90 $^{\circ}\text{C}$, giving rise to the synchronized increase of the intensity ratio of A_{2876}/A_{2867} . This result is consistent with the fact that there is the peak shift for the CH_3 stretching mode. However, the only negative peak in the difference spectra (Fig. 5a) around 2853 cm^{-1} demonstrates that there is intensity decreasing but no peak shift for the $\nu_s(\text{CH}_2)$ band.

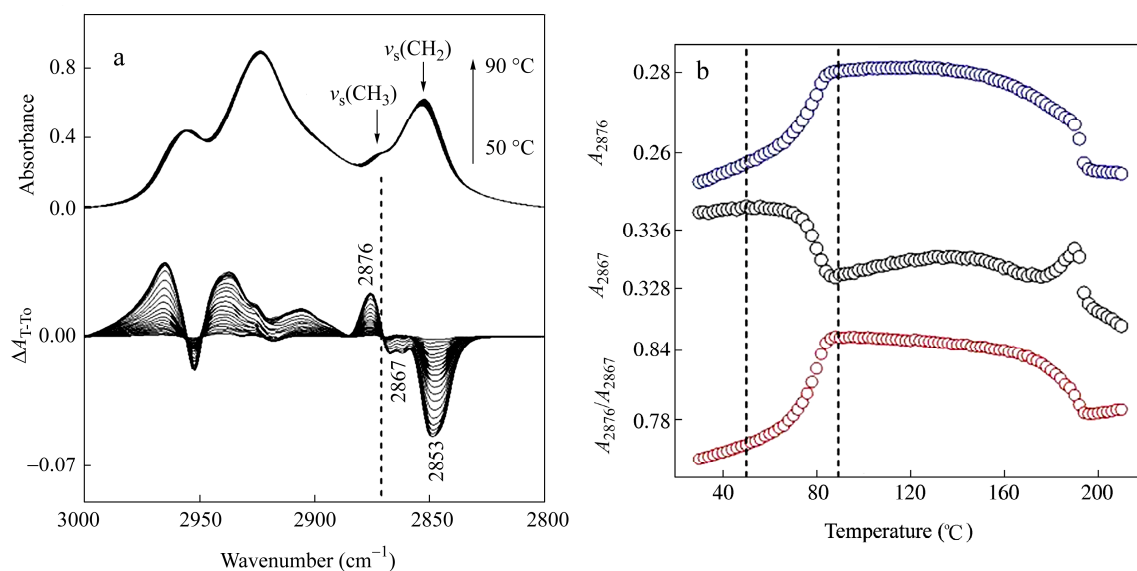


Fig. 5 (a) IR spectral changes of the solvent annealing P3OT films prepared at 10 $^{\circ}\text{C}$ in the C—H stretching vibration region from 50 $^{\circ}\text{C}$ to 90 $^{\circ}\text{C}$ and the corresponding difference spectra obtained by subtraction of the initial spectrum measured at 50 $^{\circ}\text{C}$ (shown in the up and down form, respectively); (b) Intensity changes of the bands at 2876 cm^{-1} and 2867 cm^{-1} , and their intensity ratio A_{2876}/A_{2867} as a function of temperature

Usually, the peak positions of these CH_3 and CH_2 stretching bands would shift to higher wavenumbers when the alkyl chains become more disordered. For example, the packing of poly(3-eicosylthiophene)(PEIT-HT) alkyl chains changes from the trans zigzag conformation to the disordered one accompanied by the shift in the $\nu_s(\text{CH}_2)$ band^[12]. Herein, there is only the peak shift for the CH_3 stretching mode but not for the CH_2 stretching mode. This observation can be concluded that the packing of end CH_3 groups rather than the CH_2 stems in the octyl side chains experiences an order-to-disorder process, which is associated with the endothermic peak at 87 $^{\circ}\text{C}$ of P3OT form II.

Structural change associated with conjugated backbones

Usually, the main chain conformation or the conjugated length of backbones of P3OT acts as a key factor to affect the conductivity and carriers mobility. For clarifying the effect of annealing temperature on the conjugated length, the original spectral change in the 1520–1440 cm^{-1} region is plotted on the top of Fig. 6(a). For

identifying the accurate peak positions of this band among the overlapped spectral profiles, the corresponding second derivatives of the original spectra are shown at the bottom of Fig. 6(a). The bands at 1510 cm^{-1} and 1456 cm^{-1} are assigned to the $\nu_{\text{as}}(\text{C}=\text{C})$ mode and the $\nu_{\text{s}}(\text{C}=\text{C})$ mode, respectively. Moreover, the intensity ratio A_{1510}/A_{1456} can be used to characterize the effective conjugation length or the backbone conformation of P3OT. It is obvious to find that the intensity ratio increases remarkably in the range of $50\text{--}90\text{ }^{\circ}\text{C}$ which suggests that the backbone planarity is enhanced largely in the endothermic process around $87\text{ }^{\circ}\text{C}$.

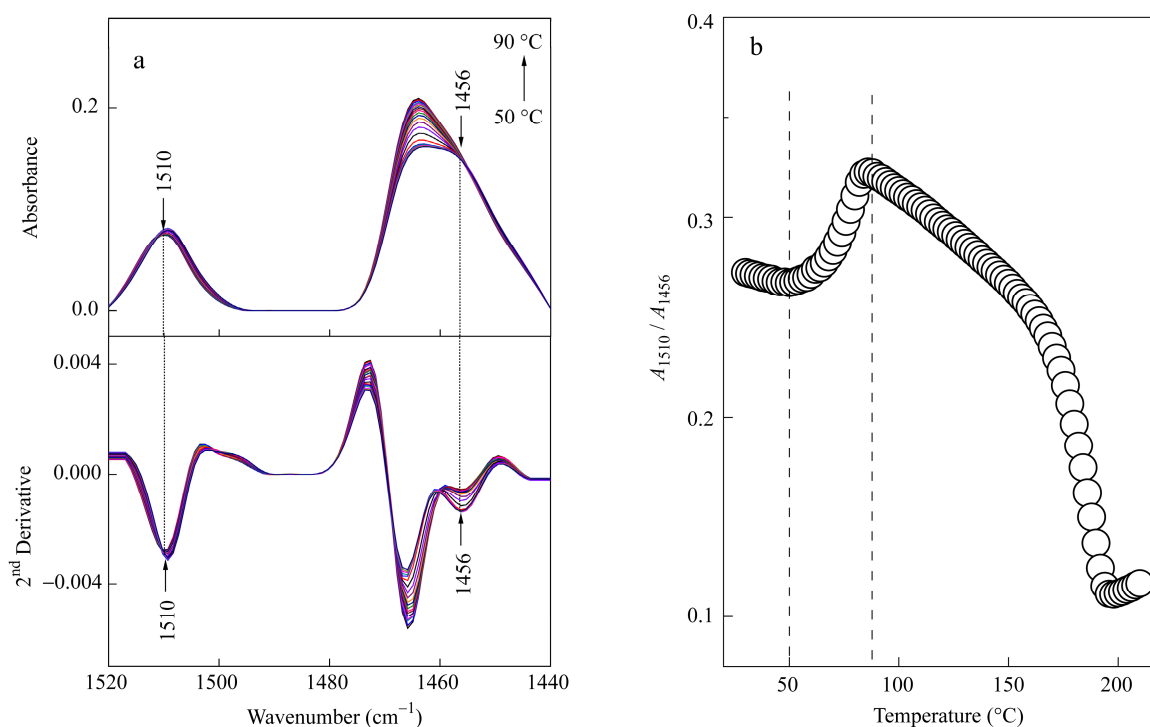


Fig. 6 (a) IR spectral changes of the solvent annealing P3OT films prepared at $10\text{ }^{\circ}\text{C}$ in the $1520\text{--}1440\text{ cm}^{-1}$ region from $50\text{ }^{\circ}\text{C}$ to $90\text{ }^{\circ}\text{C}$ and the corresponding second derivatives curves (shown in up and down part, respectively); (b) The intensity ratios of the $\nu_{\text{as}}(\text{C}=\text{C})$ to $\nu_{\text{s}}(\text{C}=\text{C})$ mode against temperature for the solvent vapor annealing P3OT films

Structural change associated with π - π stacking

Figure 7(a) displays the spectral change of thiophene $\text{C}_{\beta}-\text{H}$ out-of-plane deformation modes in the $850\text{--}800\text{ cm}^{-1}$ region, which is sensitive to the crystal modification of P3ATs. It has been reported that the band at 836 cm^{-1} is associated with the amorphous state due to its domination above the melting point. Clearly, with increasing temperature, three bands located at 826 , 820 , and 836 cm^{-1} sequentially dominate the spectral region of $850\text{--}800\text{ cm}^{-1}$. Furthermore, their intensities changes as depicted in Fig. 6(b) show that in the region of $50\text{--}90\text{ }^{\circ}\text{C}$, with the increase of the band at 820 cm^{-1} arising from form I, the intensity of the band at 826 cm^{-1} arising from form II decays and then disappears. Therefore, it is in agreement with the fact that the low temperature endothermic peak of the P3OT films annealed at $10\text{ }^{\circ}\text{C}$ is the phase transition from form II to form I. Then with temperature rising further, the intensity of the crystalline bands gradually decreases and form I starts to melt into the amorphous phase. Usually, we have previously reported that π - π interaction is greater when the wavenumber is lower. From the peak shift, it can be deduced that the increase of π - π interaction occurs during the phase transition. However, when the temperature increases gradually until the melting point, π - π interaction decreases giving rise to the blue shift of the crystal band.

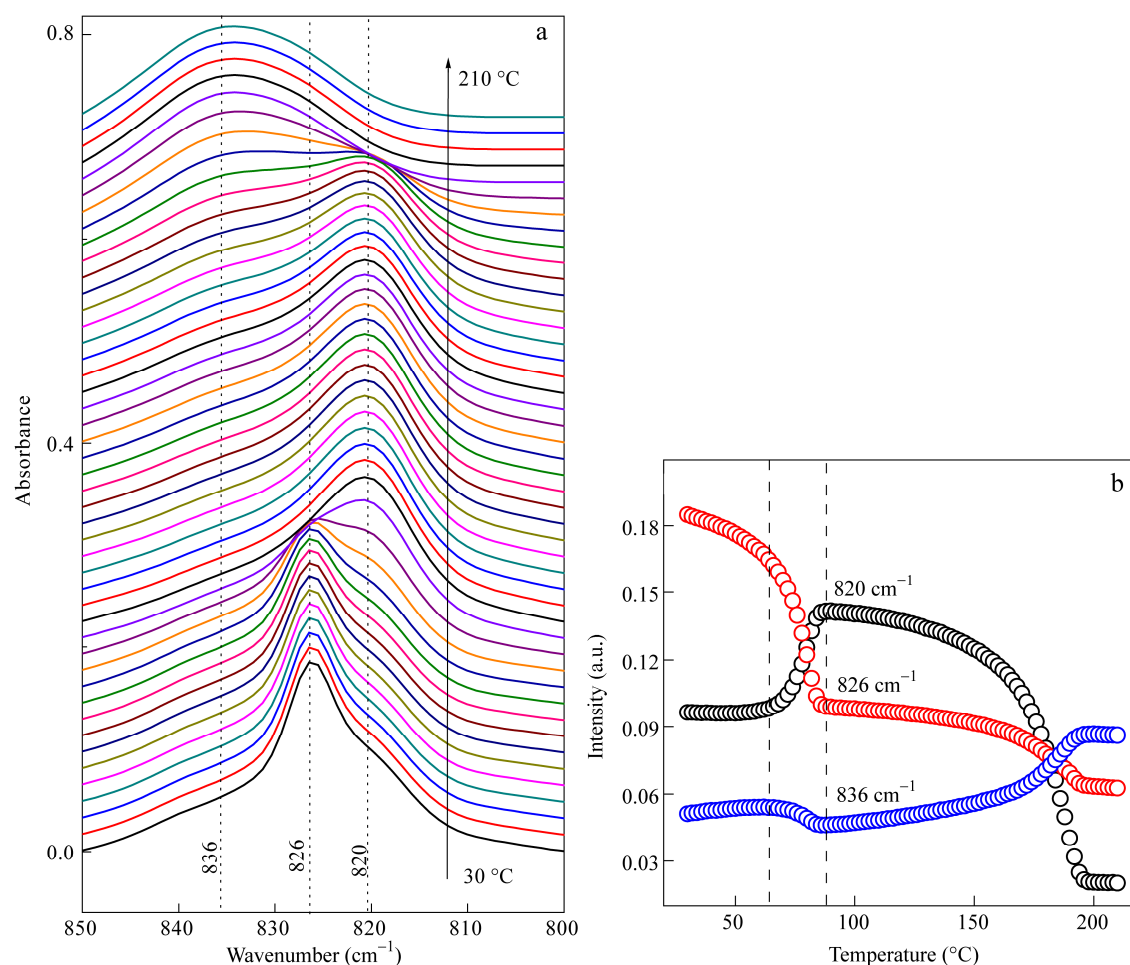


Fig. 7 (a) IR spectral changes in the $C_{\beta}-H$ out-of-plane deformation band region during the heating process of the solvent annealing P3OT films from 30 to 210 °C; (b) The change of the characteristic bands intensities as a function of temperature

By the temperature-dependent IR spectroscopy of three regions, it is clearly revealed that the low temperature endothermic behavior of P3OT can be attributed to the crystal-to-crystal (from II-to-form I) phase transition. In this phase transition process, the structural rearrangement in the chain packing modes is accompanied by the extension of the conjugated backbone and the disordering of end CH_3 groups. Moreover, $\pi-\pi$ interaction increases with this phase transition.

CONCLUSIONS

In the present study, it is found that the solvent annealing temperature makes a great effect on the crystal modifications and the phase transition behavior of poly(3-octylthiophene) (P3OT) film. When chloroform is used as the available solvent, form I and form II of the P3OT crystal modification can be respectively obtained at room temperature and relatively low annealing temperature. Comparing to the mostly used CS_2 solvent, this finding provides a more facile and environmental friend method to prepare P3OT form II. Of note, there is an obviously endothermic peak around 87 °C in the DSC heating curve of P3OT form II crystal, which can be assigned to the phase transition from form II to form I as revealed by temperature-dependent FTIR spectroscopy. Moreover, temperature-dependent IR spectra reveals that the structural rearrangement in $\pi-\pi$ packing modes together with the extension of the conjugated backbone and the disordering of end CH_3 groups could be observed during the form II to form I phase transition process.

ACKNOWLEDGEMENTS Jian-ming Zhang thanks the Alexander von Humboldt foundation for supporting his research stay at the Max Planck Institute for Polymer Research in Mainz, Germany.

REFERENCES

- 1 Bao, Z., Dodabalapur, A. and Lovinger, A.J., *Appl. Phys. Lett.*, 1996, 69: 4108
- 2 Yang, H., Shin, T.J., Yang, L., Cho, K., Ryu, C.Y. and Bao, Z., *Adv. Funct. Mater.*, 2005, 15(4): 671
- 3 Jen, K.Y., Miller, G. and Elsenbaumer, R.L., *Chem. Commun.*, 1986, (17): 1346
- 4 Sirringhaus, H., Brown, P., Friend, R., Nielsen, M., Bechgaard, K., Langeveld-Voss, B., Spiering, A., Janssen, R.A., Meijer, E. and Herwig, P., *Nature*, 1999, 401(6754): 685
- 5 Zen, A., Saphiannikova, M., Neher, D., Grenzer, J., Grigorian, S., Pietsch, U., Asawapirom, U., Janietz, S., Scherf, U. and Lieberwirth, I., *Macromolecules*, 2006, 39(6): 2162
- 6 Yang, X. and Loos, J., *Macromolecules*, 2007, 40(5): 1353
- 7 Prosa, T., Winokur, M. and McCullough, R., *Macromolecules*, 1996, 29(10): 3654
- 8 Lu, G., Li, L. and Yang, X., *Macromolecules*, 2008, 41(6): 2062
- 9 Qu, Y., Su, Q., Li, S., Lu, G., Zhou, X., Zhang, J., Chen, Z. and Yang, X., *ACS Macro Lett.*, 2012, 1(11): 1274
- 10 Yuan, Y., Zhang, J. and Sun, J., *Macromolecules*, 2011, 44(15): 6128
- 11 Yuan, Y., Zhang, J., Sun, J., Hu, J., Zhang, T. and Duan, Y., *Macromolecules*, 2011, 44(23): 9341
- 12 Tachibana, H., Hosaka, N. and Tokura, Y., *Macromolecules*, 2001, 34(6): 1823
- 13 Lu, G., Li, L. and Yang, X., *Adv. Mater.*, 2007, 19(21): 3594
- 14 Liu, J., Sun, Y., Gao, X., Xing, R., Zheng, L., Wu, S., Geng, Y. and Han, Y., *Langmuir*, 2011, 27: 4212
- 15 Causin, V., Marega, C., Marigo, A., Valentini, L. and Kenny, J. M., *Macromolecules*, 2005, 38(2): 409
- 16 Guo, Y., Jin, Y. and Su, Z., *Soft Matter*, 2012, 8(10): 2907
- 17 Guo, Y., Wang, L., Han, Y., Geng, Y. and Su, Z., *Polym. Chem.*, 2014, 5: 1938
- 18 Tashiro, K., Ono, K., Minagawa, Y., Kobayashi, M., Kawai, T. and Yoshino, K., *J. Polym. Sci. Part B: Polym. Phys.*, 1991, 29(10): 1223
- 19 Zerbi, G., Chierichetti, B. and Ingänas, O., *J. Chem. Phys.*, 1991, 94: 4646
- 20 Zerbi, G., Chierichetti, B. and Ingänas, O., *J. Chem. Phys.*, 1991, 94(6): 4637
- 21 Yazawa, K., Inoue, Y., Yamamoto, T. and Asakawa, N., *Phys. Rev. B: Condens. Matter.*, 2006, 74(9): 094204
- 22 Furukawa, Y., Akimoto, M. and Harada, I., *Synth. Met.*, 1987, 18(1): 151