

# Structure development and properties of high-speed melt spun poly(butylene terephthalate)/poly(butylene adipate-*co*-terephthalate) bicomponent fibers

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## Abstract

Ultra-high-speed bicomponent spinning of poly(butylene terephthalate) (PBT) as sheath and biodegradable poly(butylene adipate-*co*-terephthalate) (PBAT) as core was accomplished with the take-up velocity up to 10 km/min. The structure development of the individual component and the properties of PBT/PBAT fibers were investigated through the measurements on differential scanning calorimetry, wide-angle X-ray diffraction, birefringence and tensile test. Due to the mutual interaction between two polymer-melts along the spinline, the processability of both components in PBT/PBAT bicomponent spinning was improved compared with those of corresponding single component spinings. Furthermore, in PBT/PBAT fibers, the structure development of PBT component was found to be greatly enhanced, which led to the improvement in its thermal and mechanical properties; whereas the structure development of PBAT component was significantly suppressed, in which nearly non-oriented structure was observed in both crystalline and amorphous phases.

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## 1. Introduction

In bicomponent melt-spinning process, two kinds of polymers are brought in contact as separate melt streams just before the spinneret and to form a single filament with designed cross-section arrangement. Bicomponent melt-spinning has been widely applied to produce functional and novel fibers like hollow fibers, electrically conductive fibers, micro/nano fibers and thermal bonding fibers, etc. [1–4]. Furthermore, due to the mutual interaction between two polymer-melts along the spinline, suitable combination of polymers with different inherent properties can also lead to unique structure development of the individual component and thereby the enhanced structure development and/or improved processability can be obtained if compared with single component spinning [5–8,9].

In this work, we attempted to produce sheath-core type bicomponent fibers consisting of poly(butylene terephthalate) (PBT) and a biodegradable copolyester poly(butylene adipate-

*co*-terephthalate) (PBAT). It is well known that PBT fibers are applied as flexible fibers because of its reversible crystal modification between  $\alpha$  and  $\beta$  forms with the application and removal of tensile stress. PBAT fiber also exhibits elastic property with low modulus and high recoverability induced by its hard-soft segments consisting chemical structure [10]. Furthermore, the melting temperature of PBAT is about 100 °C lower than that of PBT [10]. Thus, PBAT (sheath)/PBT (core) bicomponent fibers would be an attractive material for thermal bonding nonwoven fabrics, especially when elastic property is desirable.

In addition, recently there are reports on fabrics woven by sheath-core type bicomponent fibers being composed of PBT and biodegradable polymers like poly(L-lactide), poly(butylene succinate) and poly(butylene succinate-*co*-L-lactide), etc. [11,12]. The main merit of such fabrics is that, with the presence of biodegradable component, denier-decreasing treatment can be conducted in an environmental friendly condition. Accordingly, PBAT/PBT fibers are also expected to be applied in those woven fabrics not only environmental friendly but also stretchable.

In this work, we first attempted to produce the bicomponent fiber PBAT/PBT with PBAT component as sheath and PBT

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component as core. However, in that case, the sheath of the filament remained tacky even down to the winding position, which made the filament difficult to be taken up. The tacky problem is due to the elastic nature and the sluggish crystallization kinetics of PBAT especially when processed with PBT. The latter factor will be explained below. In this work, PBT/PBAT fibers were produced by changing the PBAT component from sheath into core. Preparation of PBAT/PBT fibers will be our future work by applying cooling system along the spinline, i.e. water bath or cooling air, such methods have been successfully applied in the melt spinning of other elastic and tacky filaments [13].

In our previous study, PBAT fibers were unexpectedly found to have well-developed and highly oriented PBT-like crystal structure despite of its ideal randomness and the composition of 1:1, which was attributed to the unusual mixed crystallization behavior of butylene adipate (BA) and butylene terephthalate (BT) units. Interest of this work will be focused on investigating the effect of mutual interaction between PBT and PBAT melts along the spinline on their structure development and properties in PBT/PBAT fibers. This effect is believed to be roughly adaptable to the case of PBAT/PBT fibers in that sheath and core components are interchanged.

## 2. Experimental

### 2.1. Bicomponent spinning

Sheath-core type PBT/PBAT bicomponent fibers were produced by extruding the melts of PBT (intrinsic viscosity = 0.88 dL/g) as the sheath and PBAT (melt volume-flow rate = 3–6 ml/10 min) as the core using two different extrusion systems. Each extrusion system consists of an extruder and a gear pump. The co-axially combined polymer melts were extruded through an annular-type spinneret with a single hole ( $\phi$ 1 mm) at a temperature of 260 °C, and the mass flow rate was controlled at 3 g/min for each component. Fiber samples with take-up velocities from 1 to 10 km/min were obtained, where 10 km/min is the maximum velocity of our present winding equipment. In our previous work, the attainable highest velocities for PBT and PBAT single component spinning were 8 and 5 km/min, respectively. Therefore, the processability of both components was improved in PBT/PBAT bicomponent spinning if compared with the corresponding single component spinning.

### 2.2. Differential scanning calorimetry (DSC)

The thermal property of PBT/PBAT fibers was analyzed by using a differential scanning calorimeter. About 5 mg of fiber sample was encapsulated in aluminium DSC pan. The DSC measurement was carried out from room temperature to 250 °C at a heating rate of 10 °C/min.

### 2.3. Wide-angle X-ray diffraction (WAXD)

Wide-angle X-ray diffraction (WAXD) patterns of fiber samples were recorded by using an X-ray generator (Rigaku

Co., Japan) with a monochromatized Cu K $\alpha$  radiation and a CCD camera. The generator was operated at 40 kV and 300 mA. The camera length was 45.5 mm, and the exposure time was 25 s.

In addition to the conventional WAXD measurement, in situ WAXD measurement during the heating process was also performed. Fiber samples were set in a heating chamber with a temperature controller. The temperature was increased from room temperature to 150 °C with a heating rate of 1 °C/min. The WAXD patterns at room temperature and 150 °C were recorded.

### 2.4. Birefringence

Birefringence of the sheath and core components in the bicomponent fiber was measured using an interference microscope (Carl Zeiss Jena) equipped with a polarizing filter based on a method described elsewhere [6]. The refractive indices of the sheath and core parts  $n_{out}$  and  $n_{in}$  can be obtained using the Eqs. (1) and (2):

$$\frac{a_{out}}{A} \frac{\lambda}{2} = (n_{out} - N)(R_{out}^2 - R_{in}^2)^{1/2} \quad (1)$$

$$\frac{a_{in}}{A} \frac{\lambda}{2} = (n_{out} - N)(R_{out} - R_{in}) + (n_{in} - N)R_{in} \quad (2)$$

where  $a_{out}$  and  $a_{in}$  are the fringe shifts measured at the interface between the sheath and core, and at the center of the fiber, respectively,  $R_{out}$  and  $R_{in}$  are the inner and outer radii,  $N$  is the refractive index of immersion liquid, and  $\lambda$  is the wave length of incident light. The birefringence of the sheath and core was represented as the difference between the corresponding refractive indices in parallel and perpendicular directions to the fiber axis.

### 2.5. Tensile test

The stress–strain curves of the fiber samples were obtained using a tensile test machine (Toyosokki, UTM-4L). The gauge length was 20 mm and the tensile speed was 20 mm/min. A representative stress–strain curve was determined by at least 10 trials for each kind of fiber. Initial tensile modulus, tensile strength and elongation at break were obtained analyzing the stress–strain curves.

## 3. Results and discussion

### 3.1. Melting temperature ( $T_m$ )

DSC thermograms of PBT/PBAT fibers prepared at 1 km/min and 10 km/min were plotted in Fig. 1. The endothermic melting peaks at ca. 125 and ca. 230 °C are corresponding to the melting of PBAT and PBT components, respectively. It is notable that the melting peak of PBAT is smaller and broader compared with that of PBT. Furthermore, with increasing take-up velocity there is no distinct change in PBAT melting peaks, whereas the melting peak of PBT

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