Enhanced interfacial and mechanical property of biodegradable poly(butylene succinate) film via introducing ultrahigh molecular weight polyethylene shish-kebab fibers

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Abstract
Shear-induced ultrahigh molecular weight polyethylene fibers (UFs) with highly oriented shish-kebab structure were achieved from dilute ultrahigh molecular weight polyethylene solution. The ordered UFs were confirmed to have enough strength as reinforced filler to enhance biodegradable poly(butylene succinate) (PBS). PBS/UF composite film was prepared by solution casting and subsequent compression molding. The crystallization behavior, interface and mechanical property of the composite film were studied. Differential scanning calorimetry and x-ray diffraction measurements showed that adding UFs had little effect on the crystallinity and crystal form of PBS. But a remarkable interfacial crystallization behavior around UFs was observed in the composite film due to the unique shish-kebab structure and the relaxation of ‘kebab’. Such interfacial crystallization led to a strong interface adhesion between fibers and matrix, and finally enhanced the tensile strength and toughness of PBS. However, the Young’s modulus and yield strength of composite film decreased significantly compared to neat PBS, which was related to the break of UFs before yield point. The PBS/UF composite with such a characteristic tensile property has a huge potential for environment-friendly polymer film.

1. Introduction
Currently, the environmental pollution caused by plastic wastes has attracted considerable attention from all over the world. The synthesis of biodegradable polymers, especially for those stemming from crops, provides an appropriate solution for this issue [1–5]. Because of the reversibility of esterification, polyesters formed by condensation, ring-opening polymerization or bacterial synthesis, such as poly(α/β/γ-ester), polylactones, are the most common biodegradable polymers [4]. Poly(butylene succinate) (PBS) synthesized by succinic acid and butanediol is a typical aliphatic polyester which can be decomposed by microorganism and finally translated into water and carbon dioxide [6]. Due to the similar mechanical and thermal properties with polyethylene as well as good biodegradability and processability, PBS has a potential application in the field of environment-friendly polymer membrane. However, the practical application of PBS film is limited by its high cost and low intrinsic mechanical property.

An attractive approach to overcome the above defects is to blend PBS and natural fibers, which can not only reduce the cost but also reinforce the matrix [7–17]. These natural fibers, including flax, hemp, jute, sisal, kenaf, coir, kapok, bamboo, cellulose, sugar cane, bagasse, and cotton fibers, have been considered as the ideal fillers for biodegradable polymers because of their advantageous characteristics of low cost, flexibility and biodegradability [8]. Dorez et al incorporated cellulose, hemp, flax, sugar cane and bamboo fibers into PBS to enhance the thermostability and flammability of PBS [9]. Thirmizir et al used PBS-g-maleic anhydride as a compatibilizer in kenaf fiber reinforced PBS composite, and a slightly enhanced tensile strength was obtained.
The research of Zhou et al. suggested that the dopamine modified ramie fiber had an excellent ability of heterogeneous nucleation for PBS, and the enhanced interfacial interaction induced by interfacial crystallization was conducive to the performance improvement of matrix [11]. Tan et al. reported that the cotton stalk bast fibers in PBS matrix could act as physical hindrance to retard the movement of chain segments, which resulted in a large improvement in the storage modulus of PBS matrix [12].

Due to the poor wettability and compatibility with polymer matrix, surface modification for the natural fibers has become an essential pathway for obtaining high-performance composites [15, 16, 18-21], following with the tremendous increase of the cost for preparing composites. Polymer fibers, especially for polymer nanofibers, can avoid the surface modification procedure owing to their excellent compatibility with polymer matrix [22, 23]. Furthermore, the less loading of nanofiber means that the effect of filler on the biodegradability of matrix is minimized. Herein, polymer nanofibers are also regarded as a promising and interesting candidate for PBS reinforcement.

Shish-kebab structure induced by shear flow is a typical crystalline structure of semi-crystalline polymers, which was first observed by Pennings and Kiel [24] in sheared dilute polyethylene solution. Based on this novel microstructure, ultrahigh molecular weight polyethylene (UHMWPE) shish-kebab fibers can be spun through high shear in UHMWPE solution [24-26]. As a highly oriented structure, shish-kebab fibers are endowed with superior strength and modulus, demonstrating a potential to reinforce polymer matrices [27-30]. Compared to other polymer fibers, such as PET fiber [31] or electrospun fibers [32], the surface of UHMWPE shish-kebab fibers is scraggly due to the existence of kebab structure. This specific surface structure can not only produce an interlocking effect between fiber and matrix, but also induce the formation of interfacial crystallization (i.e. transcrysalline interphase), providing an effective way to enhance interfacial adhesion and stress transfer efficiency [33]. Nevertheless, the study concerning UHMWPE shish-kebab fiber reinforced polymer composites, especially for biodegradable polymers, has been scarcely reported.

In this work, the oriented UHMWPE fibers with shish-kebab structure were shear-induced in UHMWPE solution. The resultant fibers were then introduced into PBS matrix by solution casting and hot-pressing processes. The main aim of this paper is to explore the feasibility of directly introducing shish-kebab fibers into biodegradable polymer as reinforced filler. The tensile property and thermostability of composites were studied and the corresponding enhancement mechanism was also discussed in detail by means of interfacial crystallization.

2. Experimental

2.1. Materials
Biodegradable PBS granules (3020 MD) with a melt mass flow rate of 5 g/10 min were supplied by Showa Highpolymer Co., Ltd., Japan. UHMWPE powder with a number-average molecular weight (Mn) range of 2.0−3.0 × 10^6 g mol^{-1} was provided by Beijing No. 2 Auxiliary Agent Factory, China. Xylene (AR) used to dissolve PBS and UHMWPE in this study was purchased from Tianjin Chemical Reagent Plant, China.

2.2. Solution spinning of UHMWPE fibers
UHMEPE fibers (UFs) were prepared by using solution spinning method according to our previous work [34]. The schematic of the solution spinning setup was shown in figure 1(a). Briefly, UHMWPE powder (7 mg) was firstly dissolved completely in xylene (70 ml) at 140 °C. The resulting solution was rapidly transferred into a bath pot which was pre-heated to 105 °C in an oil bath. Subsequently, a mechanical stir bar with two symmetrical ferrum frames was introduced into the bath pot. Solution spinning was then carried out at this temperature by rotating the bar with a speed of 500 rpm for 40 min. Based on the previous study [26], the shish-kebab crystals of UHMWPE (i.e. the UHMWPE fibers, UFs) could be induced by high shear. Therefore, UFs were spun on the ferrum frames during shear-induced crystallization (figure 1(b)). The resultant UFs (the ordered shish-kebab crystals, figure 1(c)) were taken out from the solution and washed with xylene to remove amorphous polymer. Finally, the UFs were dried at room temperature for 24 h and weighted by analytical balance.

2.3. Preparation of PBS/UF composite film
PBS/UF composite films were prepared by two steps, i.e. solution casting and hot-pressing. In the solution casting process, dried UFs with ferrum frame were pre-heated to 80 °C, 20 mL PBS solution (xylene, w/v: 1/10) was then casted on UFs. After completely evaporating, a rectangular white and loose film was tailored along the edges of the ferrum frame and subsequently dried in a vacuum oven at 80 °C for 24 h. The final composite film was prepared by hot-pressing the solution-casted film via a vacuum-assisted compression machine (FM-11, Beijing Future Materials Science-Technology Co., Ltd). In detail, the resulting solution-casted film was pre-heated at 130 °C for 20 min and then pressed at a pressure of 1 MPa for 10 min, followed by fast cooling to 85 °C.
and isothermal crystallization for 30 min PBS/UFs composite film was finally obtained by cooling it to room temperature. For comparison, neat PBS film was produced in the same way. The content of UFs in PBS/UFs composite film was about 2 wt%, calculated based on equation (1):

\[ w = \frac{W_1 - W_f}{W_2} \times 100\% \]  

where \( W_1 \), \( W_f \) and \( W_2 \) are the weights of the dried fiber (with ferrum frame), the ferrum frame and the dried solution-casted film (without ferrum frame), respectively.

2.4. Characterization

The crystallization behaviors of UFs, neat PBS and PBS/UF composite were analyzed by using differential scanning calorimetry (DSC, TA Q2000) and x-ray diffraction (XRD). The samples (5–10 mg) for DSC measurement were heated from room temperature to 170 °C at a heating rate of 10 °C min \(^{-1}\) under nitrogen protection. The crystallinity \( (X_c) \) of these samples was calculated according to the following equation:

\[ X_c = \frac{\Delta H}{\Delta H_0} \times 100\% \]  

where \( \Delta H \) is the enthalpy of fusion and \( \Delta H_0 \) is the enthalpy of fusion for 100% crystallized sample (291 J g \(^{-1}\) for UHMWPE [29] and 82.3 J g \(^{-1}\) for PBS [35, 36]). The samples for XRD analysis were loaded into the sample holder (10 × 10 × 1 mm\(^3\)) of Rigaku Ultima IV x-ray diffractometer equipped with a Cu tube and a scintillation detector beam. Their XRD patterns were recorded in the 2\( ^\theta \)–70\( ^\theta \) range at steps of 0.02° with a scanning speed of 4°/min.

The morphologies of UFs and PBS/UF composite were observed using a field emission scanning electron microscope (FE-SEM, JEOL JSM-7500F) under an acceleration voltage of 5 kV. Before observation, the dried UFs and the cryo-fractured and etched surfaces of composite were sputter-coated with gold. The etching process according to our previous work [34] was carried out by soaking the composite film into xylene at 80 °C for 2 h.

Polarizing microscope (POM) was employed to better understand the interfacial crystallization behavior on a Olympus BX51 POM at room temperature. The POM sample, i.e., single-fiber bundle composite, was prepared based the following procedure: single-bundle fiber were firstly prepared through the above-mentioned spinning method with a short shear time (2 min), subsequently introduced in PBS matrix according to the process to prepare PBS/UF composite film.

Tensile properties of UFs, neat PBS and PBS/UF composite were measured on a SUNS UTM2203 universal tensile testing machine with a crosshead rate of 5 mm min \(^{-1}\) at room temperature based on the standard of ASTM D638 2003. The tensile direction of composite was along the orientation direction of UFs. At least ten specimens of each sample were tested and the results were averaged to improve accuracy.

3. Results and discussion

3.1. Structure and property of UHMWPE fibers

On the basis of previous researches [24–26, 34], UHMWPE fibers prepared via shearing polymer solution possess a shish-kebab structure, which can improve the strength of fibers due to the highly ordered polymer chains in shish. Figure 2 shows the detailed morphology of UFs observed by SEM. In this work, the amorphous
chains attached on the UFs were washed away by xylene. As shown in figure 2(a), only the expected shish-kebabs are retained after washing and orderly aligned along the shear direction. The continuous and oriented shish-kebabs are perceived as polymer fibril bundles (UFs), having the potential as reinforcing fibers [37]. The amplified image (see figure 2(b)) reconfirms the typical shish-kebab structure of UFs, consisting of a single fibrillar core and many crystalline lamellae decorated perpendicularly to the fibrillar core. The diameter of the ‘shish’ and the thickness of the ‘kebab’ are about 50 and 20 nm, respectively. Moreover, some obvious breakages (or voids) pointed by red arrows are also observed in the shish-kebabs, which provide the potential possibility for the infiltration of polymer solution. The formation mechanism of the shish-kebab by shearing the UHMWPE solution proposed by de Gennes in 1974 is widely accepted: when a shear flow is introduced in a dilute UHMWPE solution where polymer chains are generally in an equilibrium coiled state, the long coiled chains stretch along the shear direction and then crystallize into shish, the short coiled chains are subsequently adsorbed and form kebabs [38].

As a necessary condition for reinforcing fibers, UHMWPE fibers should have a strong mechanical property. For this reason, the uniaxial tensile test of UFs was carried out and the stress-strain curve was presented in figure 3. Compared to the commercial polyethylene film, the tensile property of UFs increases dramatically due to the ordered shish-kebab structure and the intrinsic oriented polymer chains in ‘shish’ [39]. In this work, the UFs with a tensile strength of 101 MPa and a Young’s modulus of 625 MPa are appropriate to enhance flexible polymer membranes. In addition, the unique shish-kebab structure of UFs can lead to a physical interlock link between matrix and fiber, which is beneficial to the improvement of interfacial interaction. Also, the rough shish-kebab structure with lots of grooves can play a role of nucleating point, resulting in the formation of interfacial crystallization. Both of them would contribute to the strong interfacial adhesion between PBS and UFs.

### 3.2. Crystallization behavior of PBS/UF composites

It is well known that adding fillers can act as heterogeneous nuclei for semi-crystalline polymers, which will affect the crystallization rate of matrix. Our previous work also confirmed that the UFs have heterogeneous...
nucleation capacity for LLDPE and significantly improve crystallization rate \cite{28}. In this study, DSC and XRD were employed to confirm the effect of introducing UFs on the crystallization behavior of PBS. The results are shown in figures 4 and 5.

As shown in figure 4(a), UFs exhibit a narrow endothermic melting peak with a melting point of 137.2 °C, which is higher than that of HDPE or LLDPE granules due to their unique shish-kebab structure (highly ordered chains) and high degree of crystallinity (78.7%). While PBS granules reveal a broad endothermic melting peak with a melting point of 126.9 °C, which may be related to its inhomogenous crystal size. In view of the melting points of pristine PBS and UFs, a compression temperature of 130 °C was selected for hot compression molding. Moreover, the processing temperature of 130 °C is just in both the endothermic peak of UFs and PBS, resulting in partial melting of both UFs and PBS, which will affect the final composite performance.

Because of the presence of self-nucleation (partial melting at 130 °C), neat PBS film shows a complex DSC melting curve with three endothermic peaks locating at 96.6, 119.9 and 139.9 °C, respectively (figure 4(b)).
According to the research reported by Satou et al [36, 40], multiple melting behavior of PBS was observed when a low cooling rate was adopted during non-isothermal crystallization or a high crystallization temperature was selected during isothermal crystallization. They attributed the multiple melting behavior to the melt-recrystallization model [40]. Wang et al studied the self-nucleation isothermal crystallization of PBS (a similar crystallization process with ours), and also found the multiple melting behavior. They explained that this phenomenon was ascribed to the different thickness of lamella formed during the self-nucleation isothermal crystallization [41], which is just appropriate for our study. Moreover, the degree of crystallinity decreases from 22.1% for PBS granules to 13.5% for PBS film in our work, consistent with the result of Wang et al [41]. After incorporating UFs, the PBS/UF composite film presents a similar DSC melting curve with neat PBS film, except the lower peak temperatures and the smaller degree of crystallinity (12.1% for PBS/UF).

Figure 5 shows the XRD patterns of neat PBS and PBS/UF composite films before and after hot-pressing. For neat PBS, the characteristic peaks with similar relative intensity at 17.6°, 19.6°, 22.6° and 24.5° assigning to (111), (002), (110) and (121) planes, respectively [42–44], are observed both before and after hot-pressing, indicating that the crystal form is not changed by the specific hot processing (self-nucleation isothermal crystallization). With respect to PBS/UF composite before and after hot-pressing, all the characteristic peaks of PBS are also found at the same diffraction angles and the characteristic peak of UFs at 21.5° assigning to (110) plane appears. However, the intensity of fiber peak reduces after hot-pressing. This result suggests that the compatibility between fibers and PBS matrix is improved during compression molding, which may be induced by interfacial crystallization [45, 46]. Based on the DSC and XRD results, it can be concluded that the introduced UFs have little effect on the crystallization behavior of PBS, at least for the degree of crystallization and crystal form [47].

3.3. Interfacial crystalline structure between UFs and PBS matrix

POM is a powerful tool to detect the interfacial crystallization morphology induced by UFs. As shown in figures 6(a) and (b), a clear interfacial crystallization phenomenon is observed around the fiber bundle, suggesting an excellent heterogeneous nucleation ability of the unique shish-kebab structure in PBS matrix. Such interfacial crystallization can not only improve the interfacial adhesion between fibers and matrix [48, 49], but also enhance the mechanical property of composite. Considering the fact that the molding temperature of 130 °C is located in the temperature range of endothermic peak of UFs, the ‘kebab’ part of the shish-kebab fibers may melt during hot-pressing (weaker stability [28]), and disappear in the resultant PBS/UF composite.

Figure 6(b) just proves this viewpoint, where the relaxant ‘kebab’ chains around the fiber bundle (black area) insert into PBS matrix. Based on these results, it can be concluded that the relaxant ‘kebab’ chains act as nucleation sites to induce interfacial crystallization, and the obtained transcristallization consisting of PBS matrix and fiber chains will further enhance the interfacial adhesion [50].
fractured surface, indicating that the oriented structure of UFs composed of the surfaces of PBS is amorphous and loose crystalline polymer chains so as to directly observe the interface between matrix and UFs because of the interfacial crystallization. Etching PBS compact coating without any voids suggests that a strong interfacial adhesion has been formed between PBS and UFs.

It is easy to (the original dense UFs, the sparser and interface regions in surface, proving the guess that the completely exposed UFs pointed by white arrow in the partial enlarged drawing of the partial enlarged drawing, which directly con-

To further confirm the enhanced interface by interfacial crystallization, the cryo-fracture and etched surfaces of PBS/UF composite film were observed by SEM. As shown in figure 6(c), aligned ridge morphology composed of the fiber bundles coated by matrix is observed in the low magnification SEM image of cryo-fractured surface, indicating that the oriented structure of UFs (see figure 2) is maintained to some extent. The high magnification SEM clearly demonstrates that the UFs are coated by PBS matrix (see figure 6(d)). The compact coating without any voids suggests that a strong interfacial adhesion has been formed between PBS matrix and UFs because of the interfacial crystallization. Etching PBS/UF film in xylene can eliminate amorphous and loose crystalline polymer chains so as to directly observe the interface between matrix and fibers. It is easy to find the semi-coated and completely exposed fibers with different etching degrees in figures 6(e) and (f), respectively. Clear interfacial crystallization regions (dotted arrows in figure 6(e)) are observed around UFs in the partial enlarged drawing, which directly confirms the existence of interfacial crystallization in PBS/UF film. The completely exposed UFs pointed by white arrow in the partial enlarged drawing of figure 6(f) show a smooth surface, proving the guess that the ‘kebab’ part of UFs is molten during hot-pressing. However, the sizes of UFs and interface regions in figures 6(e) and (f) seem to be much larger than that of the original shish structure. This is attributed to the fact that UFs tend to form bundle during spinning and compression molding. Compared to the original dense UFs, the sparser fibers appearing on the etched surface may be ascribed to that the fibrils are not substantially exposed due to incompletely etching as mentioned above.

### 3.4. Mechanical property of PBS/UF composites film

UHMWPE fibers are expected to improve the mechanical property of PBS matrix because of their higher strength and modulus and interfacial crystallization. The representative stress-strain curves of neat PBS and PBS/UF composite films are shown in figure 7, and table 1 lists the tensile parameters including yield strength, ultimate tensile strength, elongation at break and Young’s modulus. When incorporating a small amount of UFs, one can clearly see that the tensile strength and elongation at break of PBS matrix increase simultaneously by about 9.9% and 18%, respectively, but the yield strength and Young’s modulus decrease significantly by 39% and 78%, respectively. As discussed in 3.1 and 3.3 sections, the high-strength UFs and the strong interfacial adhesion, which improves stress transfer between fibers and matrix [21,51–53], are the main reasons for the enhanced tensile strength and toughness of PBS/UF composite film. However, the dramatic decreases of yield strength and Young’s modulus may be attributed to the low elongation at break of UFs (see figure 5), and the partly structure destruction of fibers during heat pressing (proved by the smooth fiber surface in figures 6(c) and (d)). Moreover, the tensile toughness of UFs in composite film would further decrease due to the relaxation of

![Figure 7. Representative stress-strain curves of neat PBS and PBS/UF composite films.](image)

| Sample          | Yield strength (MPa) | Tensile strength (MPa) | Elongation at break (%) | Young’s modulus (MPa) |
|-----------------|----------------------|------------------------|-------------------------|-----------------------|
| Neat PBS        | 9.54 ± 0.55          | 12.69 ± 0.97           | 1158 ± 123.5            | 196.3 ± 10.2          |
| PBS/UF          | 5.88 ± 0.29          | 13.95 ± 1.41           | 1369 ± 242.3            | 42.9 ± 2.5            |
| (increment)     | (−39%)               | (+9.9%)                | (+18%)                  | (−78%)                |

Table 1. Mechanical parameters obtained from stress-strain curves.
*kebab*. The possible mechanisms for the tensile behavior of PBS/UF film are described as follows: Under uniaxial tension loading, the continuous UFs in composite film firstly broke before yield point, and then some voids appeared at the breaking points of fibers, causing the decrease of actual cross-sectional area. On the other hand, the melting destruction in the structure and properties of UFs during heat pressing may also weaken their enhancement effect for the composite. Therefore, both of these reasons resulted in the significant decreases of yield strength and Young’s modulus. Even so, such micro-cracks and melting destruction of reinforced fibers did not lead to the failure of matrix because the spread of micro-cracks was prevented by the strong interface.

The melting destruction in the structure and properties of UFs during heat pressing may also weaken their strength and toughness and low stiffness makes PBS/UF composite have a huge potential for polymer film.

4. Conclusions

In this work, solution-spun UFs were fabricated by shearing dilute UHMWPE solution. SEM images reveal that the UFs are composed of highly oriented shish-kebab structure, resulting in enhancing tensile strength and modulus compared to PE films. Subsequently, the UFs as reinforced filler were used to enhance biodegradable PBS, and PBS/UF composite film was prepared by solution casting and compression molding. DSC and XRD measurements suggest that the added UFs have little effect on the crystallinity and crystal form, but a multiple melting behavior is observed in both neat PBS and composite DSC curves due to the special hot-processing temperature of 130 °C is just in both the endothermic peak of UFs and PBS, resulting in partial melting of both UFs and PBS. Furthermore, a remarkable interfacial crystallization behavior around UFs is found in the composite film. Except that the heterogeneous nucleus of UFs with the unique shish-kebab structure, the relaxation of *kebab* is also related to the formation of transcristallization. Moreover, the interfacial crystallization leads to a strong interface adhesion between fibers and matrix and finally enhanced the tensile strength and toughness of PBS. However, the Young’s modulus and yield strength of composite decrease significantly comparing to neat PBS, which was related to the break of UFs before yield point. The PBS/UF composite with such a characteristic tensile property have a huge potential for environment-friendly polymer films.

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