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Influence factors of internal structure and interfacial compatibility of UHMWPE fiber/SEBS resin composites: Processing parameters, structure of fiber and nature of resin

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ABSTRACT

This study investigates in detail the effects of processing parameters (pressure and temperature) on the internal structure and interfacial compatibility of unidirectional (UD) sheets of ultrahigh molecular weight polyethylene (UHMWPE) fiber/styrene–ethylene–butylene–styrene (SEBS) resin composites by dynamic mechanical thermal analysis (DMTA) and T-peel tests. The variation of storage moduli of the composites with the processing pressure indicate that almost all of the resin phase had changed into the interfacial phase; the $\tan\delta$ curves also testified that the ethylene–butylene (EB) segment of SEBS matrix was entangled with the molecular chains of polyethylene (PE) fibers. Moreover, the result of the T-peel tests of composites indicated that the pressure produces more significant changes than the temperature for both monofilm and no-film UD composites. Besides, the latter had higher adhesion strengths under the same processing conditions due to increased adhesion between the fiber and the resin. Next, high-density polyethylene (HDPE) modified SEBS resin was used as the matrix of the composites. Their interfacial adhesion strengths were found to increase slightly and the DMTA curves indicate that the crystallinity of the modified matrix increased with the content of HDPE resin. All of these benefit the improvement of the protection property of the composites.

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1. Introduction

High strength fiber/resin composite material is now the predominant human body protection product due to its unusual impact-resistant property [1–3]. Among these reinforcing fibers, ultra-high molecular weight polyethylene (UHMWPE) fiber has the lowest density, below 10^3 kg/m^3, and exhibits excellent energy-absorption [4,5]. This also makes it very suitable as a body protection material [6]. However, UHMWPE fiber is known to have a chemically inert surface, which leads to poor interfacial adhesion in UHMWPE fiber/resin composites, thereby greatly restricting its application. The situation is likely to become more complicated if the fiber needs surface modification. While improving its surface properties, we must ensure its mechanical properties change little [7–9].

Another possible approach is to modify the nature of matrix for the improvement of the performance of fiber/resin composites. Although the content of resin is much lower than that of fibers in terms of body-protection composites, the role of the matrix should not be neglected [10,11]. The continuous matrix in these composites plays an important role for energy absorption in the penetration direction and energy dissipation along ply direction [12]. Our previous investigation [13] indicated that the impact and cohesion strength of styrene–ethylene–butylene–styrene (SEBS) resin, a kind of thermoplastic elastomer, could be raised by the simple method of blending it with high-density polyethylene (HDPE) resin. Both SEBS resin and HDPE resin have domains similar to that of UHMWPE. Therefore, an appropriate interfacial compatibility of the composites could be obtained if HDPE-modified SEBS resin was used as the matrix of UHMWPE fiber, although a systematic study to explore the effects of processing parameters on the resulting composites has not been done.

It is believed that processing parameters have a great influence on the structures and properties of UHMWPE fiber/resin composites, but the variations of the internal structures of these composites have not yet been made clear. In this study, we systematically investigated the effects of processing parameters on the internal structure and interfacial compatibility of UHMWPE fiber/SEBS resin composites. For the convenience of preparation, two kinds of commercial unidirectional (UD) sheets of UHMWPE fiber were used, both of which have been applied widely in the body protection field.

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Furthermore, the influence of the structure of unidirectional sheet and matrix properties on the interfacial compatibility between UHMWPE UD and the resin will also be elucidated in detail.

2. Experimental methods

2.1. Materials

The UHMWPE UD composite used was UD-ZT161 (Hunan Sino-Thai Special Arming Co., China), which was made of UHMWPE fibers of roughly 20 μm diameters. This was coated with commercially available SEBS resin. The SEBS resin (Kraton G1652, Shell Chemical Co., Guangzhou, China) and HDPE resin (M80064, Saudi Basic Industries Corp., Riyadh, Saudi Arabia) were used as matrix. A commercial yarn of UHMWPE fiber (ZTX99, Hunan Sino-Thai Special Arming Co., China) was used in dynamic mechanical thermal analysis (DMTA) testing. Two kinds of UHMWPE UD were used in the study, monofilm UD and no-film UD. Both were multi-layer laminated UHMWPE unidirectional sheets, and the only difference being that there was a 0.03 mm-thick polyethylene (PE) film on one side of the former, while the latter did not. For sake of simplification, the latter was mainly discussed.

2.2. Fabrication of UHMWPE fiber/resin composites

UHMWPE fiber/resin composites were obtained using UHMWPE UD by a hot-press method. The processing temperature sequence was 110 °C, 120 °C, and 130 °C, and the processing time was 25 min. Processing pressure was 3 MPa during the initial 5 min and 6 MPa, 8 MPa, 10 MPa or other designed pressures for the rest of the processing cycle. Every specimen consisted of two plies no-film UD with a thickness of 0.5 ± 0.05 mm for Interfacial Adhesion Test (0°/0°) and DMTA (0°/90°). Similarly, the monofilm UD sheet was also used in Adhesion Test, and they were positioned face-to-back during the preparation. When HDPE-modified SEBS was studied as matrix, a layer of SEBS/HDPE film of about 0.1 mm was obtained firstly through hot-press molding. The film was sandwiched between two sheets of UHMWPE UD. The preparation and measurement of the specimens is shown as Scheme 1.

2.3. Interfacial adhesion test

Interfacial adhesion of the UHMWPE UD composites was measured by a T-peel test (ISO 11339:1993) using an Instron tensile tester (model 4465). Each specimen was 200 mm in length and 25 mm in width. The crosshead speed was 100 mm/min. At least five specimens for each sample were measured, and the average value of their T-peel strength was calculated and reported. The test sample and the experimental setup are both shown in Fig. 1.

2.4. Dynamic mechanical test

Dynamic mechanical thermal properties, polymer damping peaks ($\tan\delta$), and the storage modulus (E′) were determined using a dynamic mechanical thermal analyzer (Rheometric Scientific Inc., model IV). The typical specimen size was $30 \times 4.5 \times 0.5$ mm^3, and the data presented in this work were ran at 1 Hz using the tensile testing mode over a temperature interval of −120 to 120 °C and a heating rate of 3 °C/min. Fig. 2 is a schematic diagram of principle for DMTA measurement.

3. Results and discussion

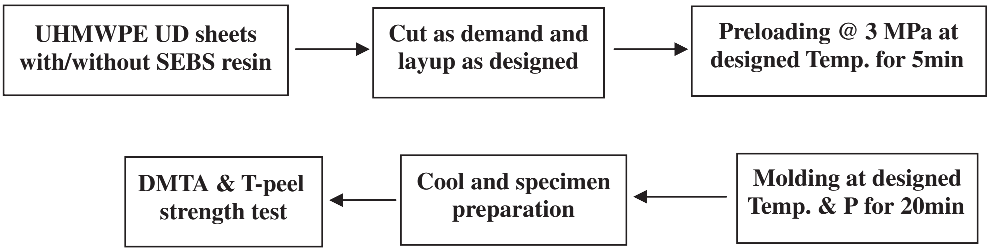
This study aims to prepare optimum UHMWPE fiber/resin composites for human body ballistic protection. In general, heating is a time-dependent heat transfer process during hot-press molding of composites such that a heterogeneous temperature field is present inside fiber/resin composites. The real temperature–time curve was recorded using prearranged thermo-sensitive probes within UHMWPE fiber composites (Fig. 3). Balance temperature for target laminates of an areal density of 5.2 kg/m^2 was achieved after 2 min. As shown, a higher molding temperature led to a higher equilibrium temperature and a longer time needed to achieve equilibrium under the same pressure. Moreover, the processing pressure also had some influence on heat transfer. A higher processing pressure led to a higher rate of heat transfer and a shorter time needed to achieve equilibrium.

To gain fundamental understanding of the subject, the first investigation deals with the effects of processing pressure (load applied during composite processing) and temperature on the internal structure of UHMWPE fiber/SEBS resin composites.

3.1. Effect of processing parameters

Dynamic mechanical testing is an effective tool to study compatibility of different components in composites [14]. The position and strength of the loss peak ($\tan\delta$) can provide information on the interaction between the fiber and resin at the molecular level. There were two primary transitions for SEBS matrix: β_r relaxation process assigned to the glass transition for the EB segment (−40 °C) and α_r relaxation process assigned to the glass transition for the polystyrene segment (100 °C) (Fig. 4a). Three significant transitions for UHMWPE fiber are also shown in Fig. 4a, namely a strong transition attributed to the α_f process assigned to PE melt transition, a transition attributed to the β_f process (40–80 °C, discussed in more detail later), and a weak signal for the γ_f relaxation process for ethylene chains at about −40 °C. The last partially overlapped with the β_r process of the SEBS resin. In addition, storage modulus (E′) of SEBS resin and UHMWPE fiber as a function of temperature are shown in Fig. 4b, respectively. The E' of the SEBS resin was several orders of magnitude lower than that of the UHMWPE fiber. This indicates that the degree of crystallinity and molecular weight of the UHMWPE fiber were far higher than those of the matrix.

The influence of the processing parameters on the structures and properties of UHMWPE fiber/resin composites can be explained by molecular structural reasons. Because the content of UHMWPE fiber in the composites was about 80 wt.%, the $\tan\delta$ of the composites reflected mainly the characteristics of UHMWPE



Scheme 1. Preparation and test of UHMWPE Fiber/SEBS resin composites.

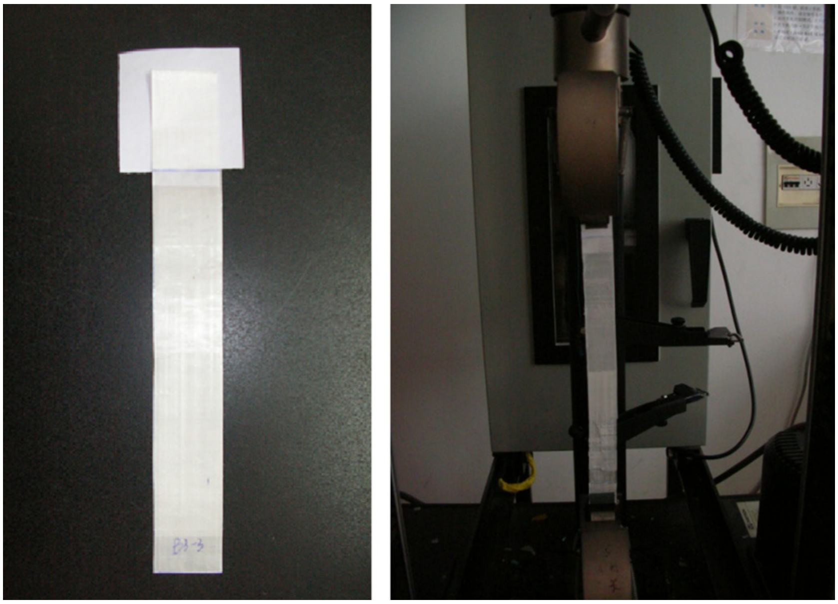


Fig. 1. The test sample (left, before testing) and the experimental setup for T-peel test (right, 180-degree peel).

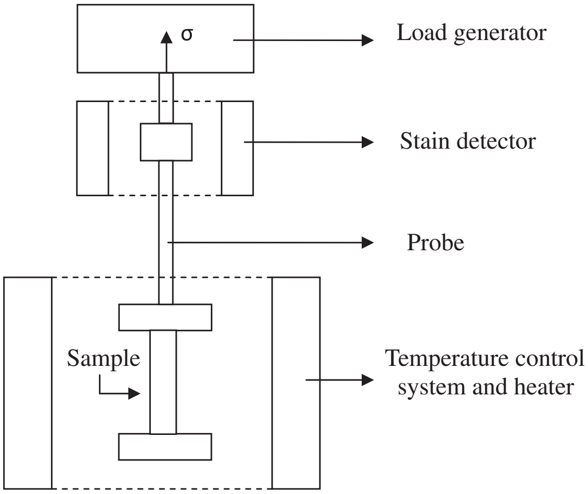


Fig. 2. Schematic diagram for tensile mode of DMA test.

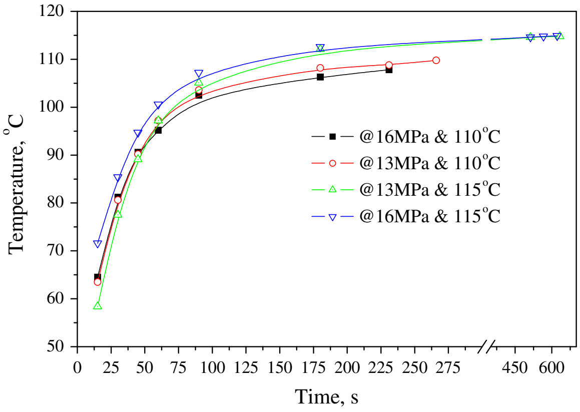


Fig. 3. Heating-up curves of lamination with hot-press method when pressure and temperature were fixed as shown (laminated by UHMWPE UD sheet, 0°/90°, without any film on two sides, areal density: 5.2 kg/m^2).

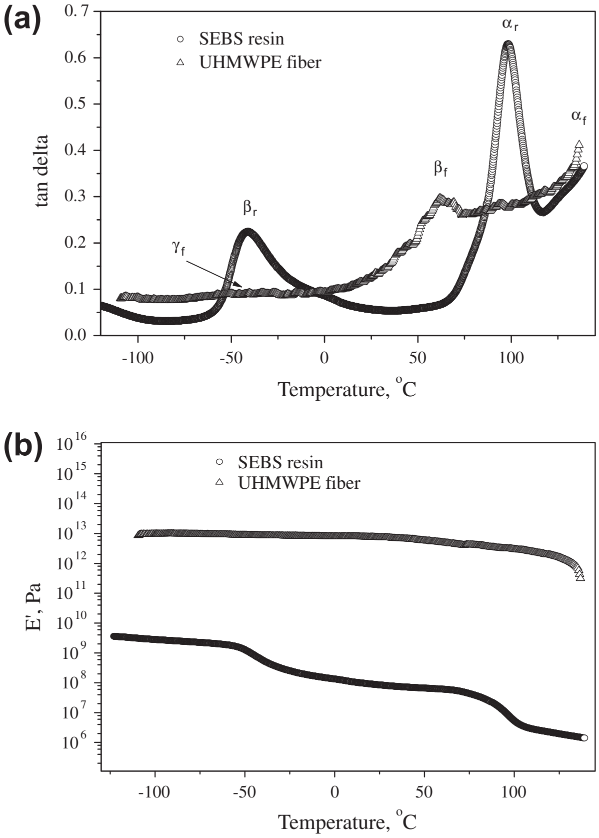


Fig. 4. Dynamic tensile response (E' and tan delta) *versus* temperature for SEBS resin (Kraton G1652, Shell Chemical Co., Guangzhou, China) and UHMWPE fiber (ZTX99, Hunan Sino-Thai Special Arming Co., China), respectively.

fibers with crystallization beyond ~95%. This includes the melting relaxation process above 100 °C, the β process from 40–80 °C, and the almost absent γ process near −40 °C. The effects of various processing pressures were investigated at a fixed temperature, and the effects of various processing temperatures were investigated at a fixed pressure.

The tan δ curves for UHMWPE UD with three pressure levels at 110 °C are presented in Fig. 5a. The strength of the β process increased with increasing processing pressures and as the position of the loss peak moved to higher temperatures. This is because the mechanical β process involves the softening or deformation of the amorphous component [15]. The relaxation of the amorphous fraction is the result of the translational mobility of the crystal lamellar coupled to the amorphous fraction. The translational component of the motion leads to the reorganization of the crystal surface, hence the redistribution of tight and loose folds and the cilia and intercrystalline links that make up the amorphous layer [16]. The higher relaxation strength of the β process means that the increase in processing pressure resulted in more amorphous components in the composite crystals, especially when the pressure reached 10 MPa. At the same time, translational movement of the crystal lamellar of PE became harder because of molecular entanglement between the resin matrix and the PE fiber and the decrease in free volume.

In contrast, for UD fiber-reinforced composites, the storage modulus in the fiber direction can be expressed as:

$$E'_c = E'_f V_f + E'_m V_m + E'_i V_i \tag{1}$$

where E' refers to the storage modulus; V is the volume fraction; and subscripts c, f, m, and i refer to the composites, fibers, matrix, and interface respectively. However, the storage modulus in the direction perpendicular to the fiber depends to a large extent on the lowest modulus of the fiber, matrix, and interface phases.

A property of UHMWPE composite laminates with cross-ply (0°/90°) that distinguishes them from other composites is that the contribution of their storage modulus comes partly from the lowest modulus of all phases because the modulus in two mutually perpendicular directions are the same. At the same time, the fiber phases are also partly contributory.

In general, the modulus of the matrix is higher than that of the interfacial phase for fiber/resin composites. This is not the case for the UHMWPE fiber/SEBS resin composites. Since the molecular chains of SEBS resin can be integrated easily with the crystal lamellar on the surface of the UHMWPE fiber under higher pressure, the E' of the interfacial phase was higher than that of the matrix phase. As shown in Fig. 5b, the E' of the UHMWPE fiber/SEBS resin composites manufactured at 6 MPa under room and low temperature was closer to that of the resin. However, its E' greatly increased with an increase of processing pressure. This implies that almost all of the resin phase had changed into the interfacial phase, which has a higher modulus, via high pressure processing. The E' of the composites rapidly declined when the testing temperature exceeded 40 °C and even overlapped with the E'–T curve of the 6 MPa processing when the testing temperature exceeded 75 °C. Variation of the dynamic modulus of the composites *versus* temperature also showed that a crystalline β process existed at 40–80 °C. Therefore, the increase in the E' of the composites obtained at high pressure came mainly from some macromolecular crystal defects.

Fig. 6a shows the tan δ curves for UHMWPE fiber/SEBS resin composites at three temperature levels under the same processing pressure of 10 MPa. The loss of strength of the β process declined with an increase in the processing temperature, such that the peak disappeared almost completely when the temperature exceeded

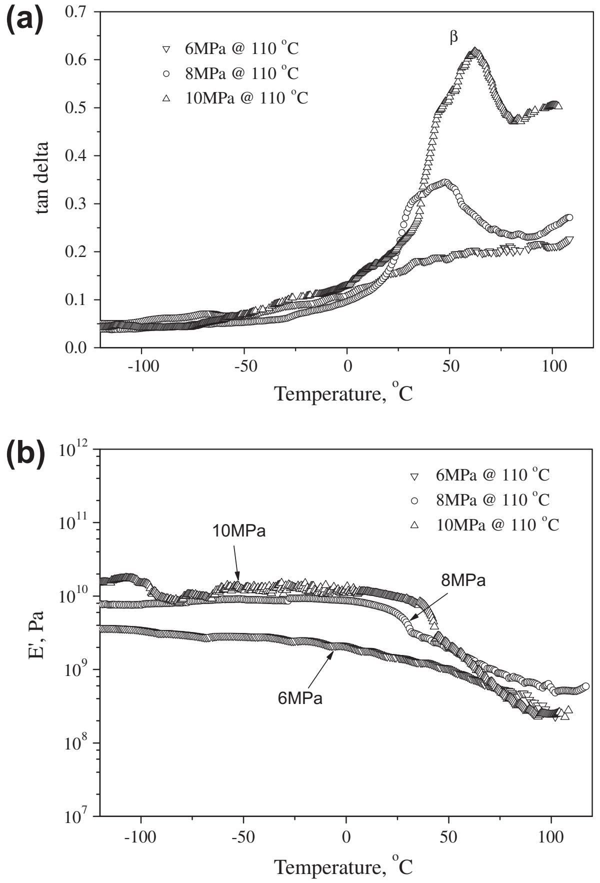


Fig. 5. Dynamic tensile response (E′ and tan delta) *versus* temperature for as-received UHMWPE UD sheet (UD-ZT161, about 80 wt.% fiber, without any film on two sides, two plies, 0°/90°) after hot-processing at three different pressures.

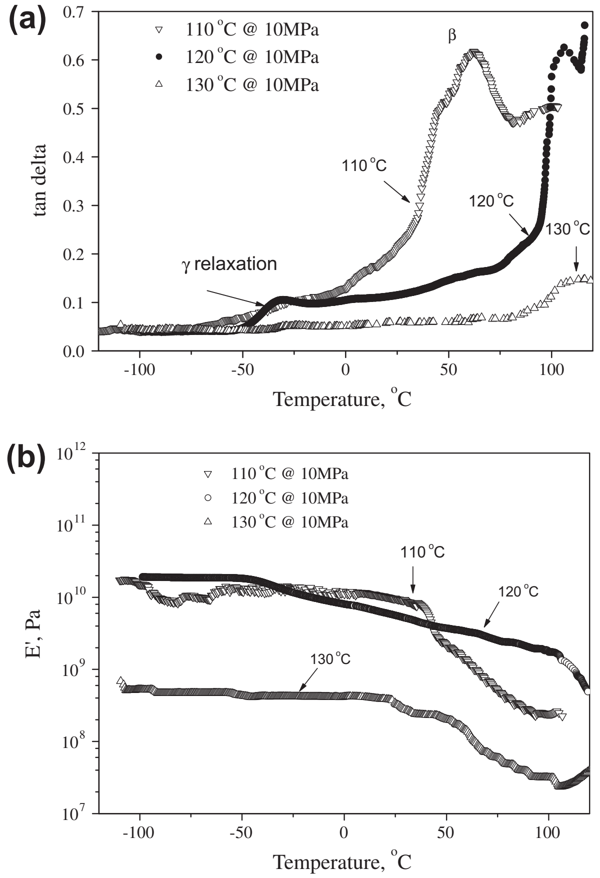


Fig. 6. Dynamic tensile response (E′ and tan delta) *versus* temperature for as-received UHMWPE UD sheet (UD-ZT161, about 80 wt.% fiber, without any film on two sides, two plies, 0°/90°) after hot-processing at three different temperatures.

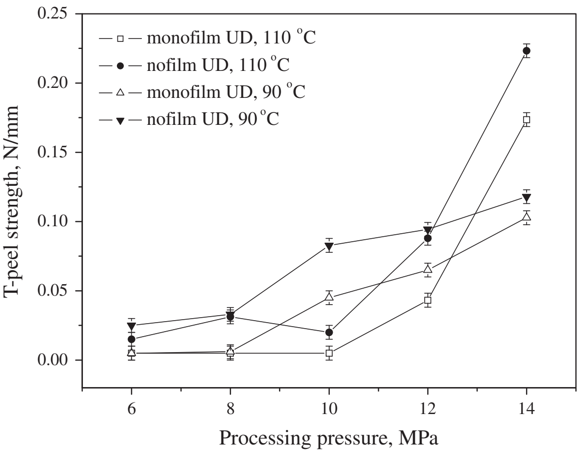


Fig. 7. Effect of processing pressure and temperature on the T-peel strength of two kinds of as-received UHMWPE UD sheet (monofilm UD: with PE film of 0.03 mm thick on one side and nofilm UD: without any film on two sides; 0°/0° layup and two plies).

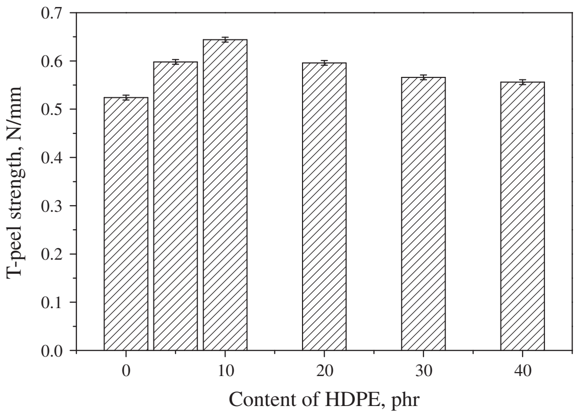


Fig. 8. Effect of content of HDPE resin on T-peel strength of HDPE-modified SEBS/UHMWPE-UD composites (UD-ZT161, 0°/0°, two plies, the content of UD: 75 wt.%; a SEBS/HDPE film of about 0.1 mm thick was used as modified matrix and sandwiched between two UHMWPE UD sheets).

120 °C. This is because the EB segment of the SEBS matrix and the molecular chains of PE fibers entangle with each other more easily during hot processing, especially when the processing temperature was near the melting point of PE fibers. This resulted in a small probability for the formation of the mobile localized structure (i.e., smooth twist) in the composites.

Because the moduli of the UHMWPE fiber and the SEBS resin differ by several orders of magnitude, the matrix phase and interfacial phase, which have lower moduli, are the two primary factors in determining the dynamic storage modulus of UHMWPE fiber/SEBS resin composites. However, the role of the fiber phase cannot be ignored when the modulus of UHMWPE fiber decreased sharply. Fig. 6b clearly shows that the modulus of UHMWPE UD decreased greatly when the processing temperature was 130 °C, even lower than that of the SEBS resin. This suggests that the crystal of the SEBS matrix had also been destroyed. Furthermore, the plots of loss strength of the γ process in UHMWPE fiber composites imply that the origin was largely amorphous with some crystalline contribution.

The interfaces between the UHMWPE fiber and the matrix have a great influence on the adhesion properties of a composite. If there is insufficient adhesion between the fiber and the matrix, the composite may become prone to delaminate when impacted by bullets, resulting in a loss of strength. Thus, the T-peel test of the composites was used to explore adhesion properties. Fig. 7 shows the changes of T-peel strength of UHMWPE fiber/SEBS composites with varying processing pressures and temperatures using two different UHMWPE UD sheets.

The T-peel strength of the UHMWPE fiber/SEBS composites increased with increasing processing pressures, regardless of processing temperature. However, a greater increase in adhesion strength can be achieved when pressure is up to 12 MPa at a high temperature (110 °C). In contrast, the no-film UD composites had higher interfacial adhesion strengths than those of the monofilm

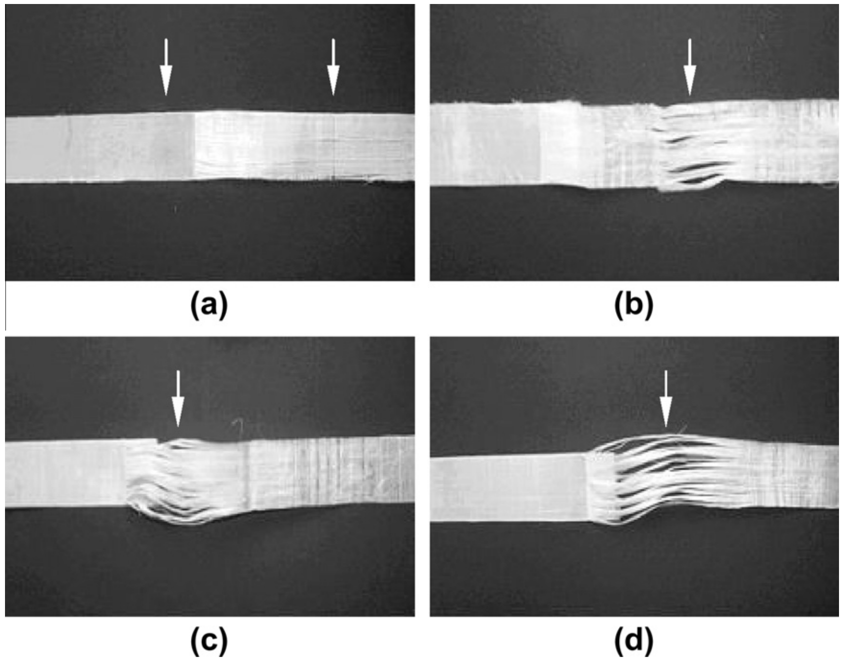


Fig. 9. Failure modes of UHMWPE UD/SEBS/HDPE composites having the weight ratio (a) 300/100/0; (b) 300/91/9; (c) 300/83/17; (d) 300/71.5/28.5 after T-peel test (UD-ZT161, 0°/0°, two plies; a SEBS/HDPE film of about 0.1 mm thick was used as modified matrix and sandwiched between two UHMWPE UD sheets).

Table 1
Results of T-peel test of HDPE-modified SEBS/UHMWPE UD composites (the content of the UD sheet: 75 wt.%; the weight ratios of HDPE *versus* SEBS were shown as below, a SEBS/HDPE film of about 0.1 mm thick was used as modified matrix and sandwiched between two UHMWPE UD sheets).

HDPE/SEBS (weight ratio)	Failure pattern			T-peel strength (N/mm)
	Adhesive failure (AF)	Cohesive failure (CF)	Delamination (layers)	
(a) 0/100	100	0	0	0.524
(b) 10/100	20	60	20	0.624
(c) 20/100	30	52	18	0.596
(d) 40/100	50	36	14	0.556

UD composites. This implies that the increase in adhesion strength is mainly because of physical wetting between fibers and resins under lower temperature, which occurs when high processing pressure brings the PE fiber and SEBS resin even closer together. Moreover, adhesion strength had a noticeable increase under higher heat and pressure because of the simultaneous recrystallization of the UHMWPE fiber and the EB segment of SEBS.

3.2. Effect of properties of the matrix

In a previous study, the impact and tensile properties of the SEBS matrix have been proven to be obviously improved by the incorporation of HDPE resins [13]. In this study, the role of the HDPE resin in SEBS matrix for UHMWPE-UD composites was further studied.

First, the interfacial compatibility of the composites was studied by T-peel tests (Fig. 8). Compared with unmodified SEBS/UHMWPE fiber composites, the interfacial adhesion strength of HDPE-modified SEBS/UHMWPE-UD composites was slightly increased. The interfacial adhesion strength of the former was 0.53 N/mm, whereas the latter achieved 0.64 N/mm when 10 phr (parts by weight of ingredient per 100 parts of SEBS resin) HDPE was present in the matrix, showing a 21% increase. This indicates a significant improvement in the interfacial state between the UHMWPE fiber and the thermoplastic matrix after adding a small amount of HDPE resin. Previous studies have shown that the fluidity of HDPE-modified SEBS resin matrix is better than the unmodified, such that more matrices can enter the gaps between fibers [13].

The failure patterns of the T-peel test for fiber/resin composites include mainly substrate failure, cohesive failure, and adhesive failure. Although all three failure modes of bonded joints were present (Fig. 9), there were still some changes observed in the HDPE-modified SEBS matrix. Fig. 9a shows a photograph of unmodified SEBS/UHMWPE fiber sample, which is relatively smooth and flat, and without fiber entanglement. From Fig. 9b–d, more and more PE fibers were drawn out and broken apart as the HDPE resin content increased. These are typical characteristics of cohesive substrate failure. This shows that a higher and higher of adhesion strength between the matrix and the fiber exists.

Table 1 lists the results of the T-peel test of HDPE-modified SEBS/UHMWPE-UD composites. Partly cohesive delamination failure appeared in the modified composites, and the total proportion of the two kinds of failure modes was highest when the content of HDPE resin was 10 phr. This agrees with the results of past studies [13].

A series of experiments were given to exploring the structure information and dynamic mechanical properties of the above-mentioned composites. Fig. 10 shows the DMTA results of samples with varied HDPE resin contents. As can be seen in Fig. 10b, the modulus of the samples greatly increased with an increase in HDPE resin in the matrix. However, the β relaxation process at about −40 °C gradually shifted to a higher temperature zone. In Fig. 10a, the strength of the β relaxation peak also decreased as the contents of HDPE resin increased. These suggest that the crystallinity of the modified matrix increased as the contents of HDPE resin increased and that the activity of HDPE, UHMWPE, and SEBS chains hardened. Since chains become more entangled, the β relaxation process requires more energy.

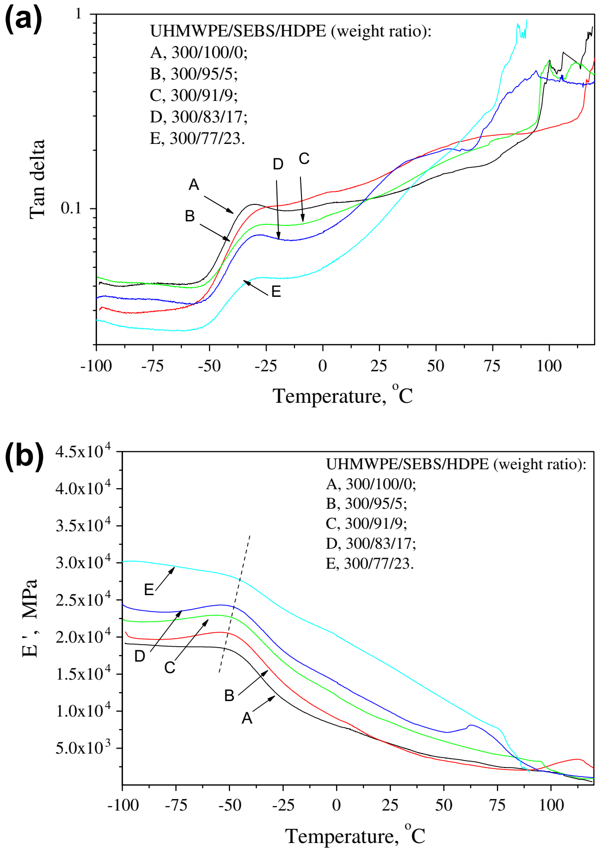


Fig. 10. Dynamic tensile response (E′ and tan delta) *versus* temperature for HDPE-modified SEBS/UHMWPE-UD composites of different HDPE resin contents (a SEBS/HDPE film of about 0.1 mm thick was used as modified matrix and sandwiched between two UHMWPE UD sheets, 0°/90°).

4. Conclusion

Kinds of important factors that influence the internal structure and the interfacial compatibility of UHMWPE UD/SEBS resin composites were investigated in detail, including processing parameters (pressure and temperature), the modification of matrix, and the structure of UD sheets. First, the curves of DMTA showed that the relaxation strength of the β process increased with increasing processing pressures, especially at 10 MPa. Moreover, the variation of storage moduli of the above-mentioned composites with the processing pressure indicate that almost all of the resin phase had changed into the interfacial phase, which have a higher modulus, via high pressure processing. On the other hand, the tan δ curves for UHMWPE fiber/SEBS composites at three temperature levels show that the strength of the β process also changed with processing temperature and gave evidence for the entanglement of the EB segment of SEBS matrix and the molecular chains of the PE fibers.

The interfacial adhesion properties of UHMWPE fiber/SEBS composites were tested by T-peel tests. The results show that the pressure produces more significant changes than the temperature for both monofilm and no-film UD composites. Besides, the latter had higher adhesion strengths than the former under the same processing conditions.

The second part of this work studied the effects of the matrix properties on the structure and adhesion properties of the UHMWPE UD/SEBS resin composites. Compared with unmodified composites, the interfacial adhesion strength of the composites with modified matrix slightly increased, which contributes to the increase in bullet-proof of the composites. All ballistic data will be discussed in another paper. Furthermore, the DMTA curves indicate that the crystallinity of the modified matrix increased with increasing HDPE resin content and that the activity of molecule chains of HDPE, UHMWPE, and SEBS decreased, accordingly.

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