

# Single Polymer Composites by Partially Melting Recycled Polyamide 6 Fibers: Preparation and Characterization

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**ABSTRACT:** Recycled polyamide 6 (PA6) cloth was used as the only raw material to prepare single polymer composites (SPC) by partially melting the PA6 fibers. During the process, part of PA6 fibers was melted to fill the gap between the remained fibers at a temperature in the melting temperature range of fibers. The matrix was *in situ* formed by recrystallization of the molten part and bonded the remained unmolten fibers together with cooling. Effects of a critical parameter, processing temperature (222–228°C), on the partially melting behavior of PA6 fibers and the properties of the resulting SPC were

investigated by differential scanning calorimetry (DSC), scanning electron microscope (SEM), and tensile tests. As a result, at the processing temperature of 226°C, 31% of PA6 fibers were exactly melt to form the matrix and the tensile strength of prepared SPC achieved a maximal value of 138 MPa that was 105% higher than the one of neat PA6. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 118: 3357–3363, 2010

**Key words:** single polymer composites; polyamide; partially melting; recycling

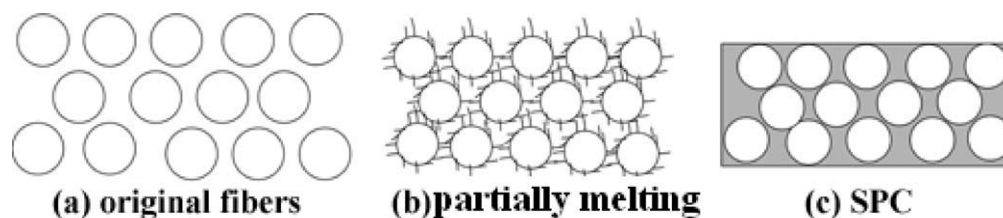
## INTRODUCTION

Since the middle of last century, composites had been widely applied in various fields, such as automobile, aerospace, and architecture, contributing to their excellent performance. However, with the aggravation of environment destruction, people made higher demands to the recyclability of used materials in all fields. For the case of fiber-reinforced polymer composites, it is hard to fully recycle this kind of composites as a result of different compositions between fiber and matrix. Recently, single polymer composites (SPC) consisting of polymer fiber-reinforced polymer matrix of same compositions<sup>1</sup> were of considerable scientific interest owing to better recyclability and interface performance than traditional glass or carbon fibers reinforced polymer composites.

Many methods, including (1) film stacking,<sup>2–4</sup> (2) powder impregnation,<sup>5,6</sup> (3) solution impregnation,<sup>7,8</sup> and coextrusion,<sup>9,10</sup> were applicable to prepare SPC. What is more noteworthy is that a special “hot compaction” process had been developed to manufacture SPC by Hine and Ward in the university of Leeds.<sup>11,12</sup> In addition, a similar process method for SPC was also investigated by other researchers<sup>13,14</sup>

and named “high-temperature high-pressure sintering.”<sup>15,16</sup> On the whole, this kind of special process method had some fundamental features that only polymer fiber was used and the matrix was formed by partially melting fibers during the process. First, regular arrays of polymer fibers were constrained by lateral pressing or radial winding for preventing high-orientation fiber molecules from relaxing with heat, as shown in Figure 1(a). At a precise temperature in the melting range of polymer fiber, part of fiber (most likely the surface having more free volume or lower degree of crystallinity) was melted to fill the original gap between fibers in a few minutes. Meanwhile, the internal part of fiber that is represented as smaller circular in Figure 1(b) was not melted and retained the intrinsic high orientation, because it had less free volume and higher melting temperature than fiber surface. This processing step of partially melting was followed by slowly cooling, during which the molten part that is represented as short lines around the circulars in Figure 1(b) began to recrystallize to form the matrix and bonded the remained fibers together. Finally, the unmolten part of polymer fibers reserving the high-orientation acted as the reinforcement phase and the recrystallized part after melting as the matrix in the obtained SPC, as shown in Figure 1(c). In a word, partially, but not completely, melting the oriented fiber phase determines the molecular continuity between the fiber/matrix interface and a narrow processing temperature window<sup>17</sup> for this process method. For the

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**Figure 1** Diagram of preparing SPC by partially melting polymer fibers.

sake of simplicity and uniform expression, this kind of process method described above is named “partially melting” in this article.

From then on, different thermoplastic polymers, including PE,<sup>18–25</sup> PP,<sup>26–31</sup> PET,<sup>32,33</sup> and PMMA,<sup>34</sup> were exploited to manufacture SPC by partially melting. Polyamide (PA) is a widely applied general plastic with high strength and good fatigue resistance. However, only the preparation of SPC from PA66 fiber by the method of partially melting had been reported<sup>35</sup> and no work about preparing SPC from PA6 fiber had been done. Therefore, in this study, PA6 fiber was firstly used to make SPC (SPC<sub>PA6</sub>) by the method of partially melting. In addition, the only raw material, PA6 fiber, was recycled from the used cloth, which improved the environmental benefits of the method of partially melting. It was concluded by other researchers<sup>33,36</sup> that precisely controlling the processing temperature is crucial to prepare SPC successfully by this method. Therefore, effect of processing temperature (222–228°C) in the melting temperature range of PA6 fiber on the partially melting behavior of fiber was investigated mainly by DSC. Observation on the fracture morphology of SPC<sub>PA6</sub> cross section by SEM showed a complete consolidation structure of composites. Comparison in tensile property of SPC<sub>PA6</sub> prepared at different temperatures displayed a balance between retaining high-orientation of PA6 fiber and forming a continuous fiber/matrix interface by partial melting.

## EXPERIMENTAL

### Materials

PA6 plain cloth was recycled from the ordinary costuming in Shanghai Sanchuan Printing and Dyeing Co. To get rid of the sizing agent on fiber surface, PA6 cloth was extracted by acetone for 24 h and then vacuum dried at 50°C for another 24 h. After the treatments, the obtained cloth was stored in a desiccator.

### Preparation of SPC<sub>PA6</sub>

Ten layers of pretreated PA6 cloth were placed into a matched steel mold that was already heated to the

designed temperature (222, 224, 226, 228°C, respectively) in a hot-press machine. This was followed by immediately applying a pressure of 5 MPa onto the mold for constraining the fibers from relaxing. Under the condition of constant temperature and pressure, it took 5 min to melt part of PA6 fibers and fill the gap between the remained fibers. Then, the whole molding system was slowly cooled to the ambient temperature in the hot-press with keeping the pressure of 5 MPa constant during this cooling process before the SPC<sub>PA6</sub> sheets were taken out. The neat PA6 sheet as control was achieved by treating the PA6 cloth under the same conditions as before except that the processing temperature was designed as 250°C for the purpose of assuring fully melting PA6 fibers. All sheets were stored in a desiccator for later tests.

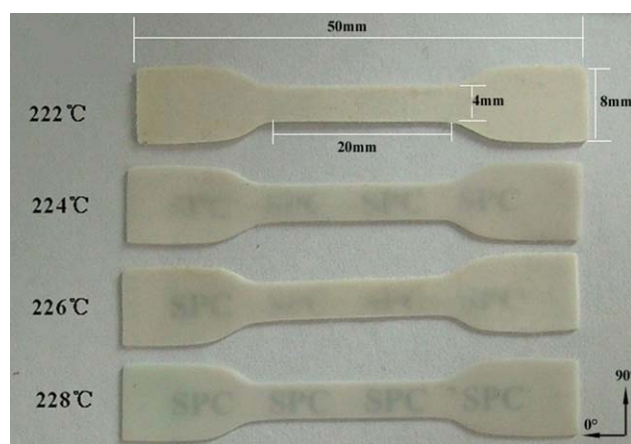
### Characterization

Four SPC<sub>PA6</sub> samples for thermoanalysis were heated from 50 to 250°C at 10°C/min under nitrogen on a Perkin–Elmer Pyris Diamond DSC instrument. The crystallinities of all samples were calculated by the theoretical crystal enthalpy of 230 J/g. Densities of SPC<sub>PA6</sub> sheets were measured according to ASTM D792. Tensile tests were performed on the dumbbell specimens (the detailed size is shown in Fig. 2) at a crosshead speed of 5 mm/min on a universal testing machine (SANS, Shenzhen, People’s Republic of China). Each test was repeated at least five times to obtain the average. Fiber/matrix interface morphologies in SPC<sub>PA6</sub> after tensile fracture were observed by SEM (JEOL, Tokyo, Japan).

## RESULTS AND DISCUSSION

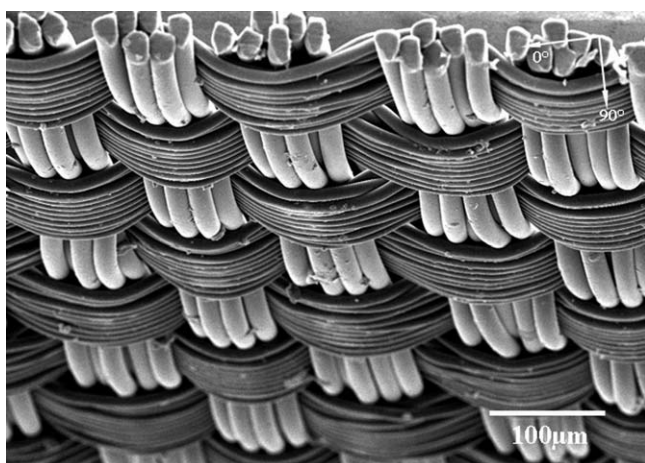
### Properties of PA6 fibers

It is needed to point out that the common PA6 cloth in clothing industry was recycled and used as raw material for its easily acquiring to embody the feasibility of preparing SPC<sub>PA6</sub> based on the partially melting behavior of PA6 fibers in this study. The fabric structure of PA6 plain cloth is shown in Figure 3 (the cloth is tilted to some extent in SEM to better display the structure).

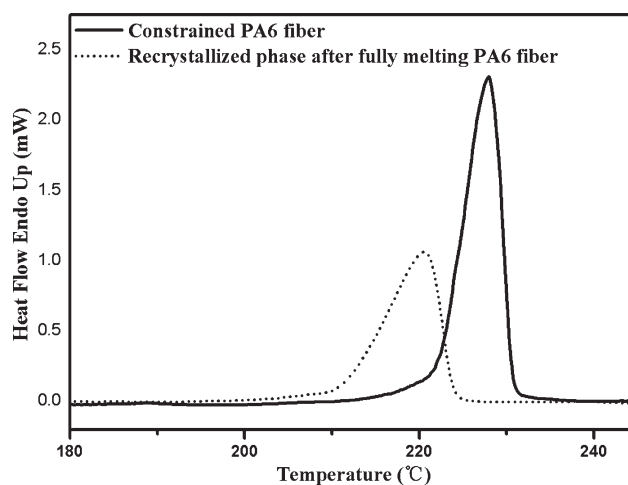


**Figure 2** Pictures of four  $\text{SPC}_{\text{PA6}}$  dumbbell specimens for tensile test by a digital camera. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]

To choose the appropriate processing temperatures in the melting range of PA6 fibers, the constrained PA6 fibers were heated from 50 to 250°C at 10°C/min and kept at 250°C for 5 min to fully melt the PA6 by DSC. Following this isothermal operation, sample was cooled to 50°C at 10°C/min for completing the recrystallization and then heated again to 250°C at the same rate. The obtained melting curves of constrained PA6 fibers and recrystallized phase after fully melting are shown in Figure 4 and the crystallization and other performance parameters of PA6 fibers are given in Table I ( $M_n$  and  $M_w$  are the number-average and weight-average molecular weight of PA6 fibers, respectively). For the purpose of constraining these monofilaments on DSC, the PA6 monofilaments that were drawn from the cloth were wound around an aluminum pan lid and two ends were tied tightly according to Ref. 36. It is necessary to mention that this constraining method of radial winding is different from the practical used method of lateral pressing during the



**Figure 3** Fabric structure of PA6 plain cloth.



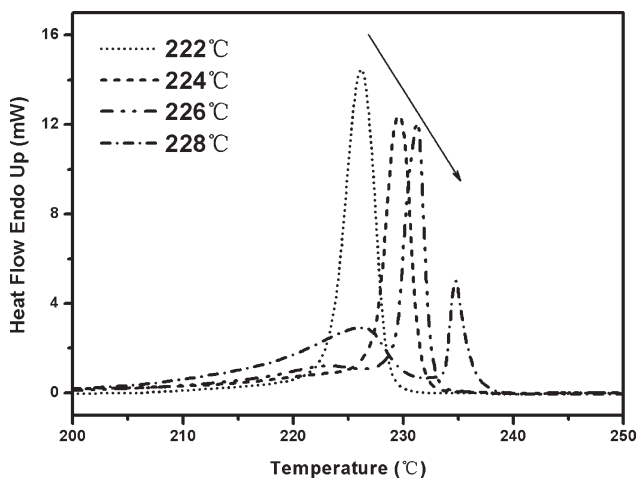
**Figure 4** Comparison of melting behaviors between constrained PA6 fiber and recrystallized phase after fully melting PA6 fiber.

preparation of  $\text{SPC}_{\text{PA6}}$ . This difference would likely result in a slight change of PA6 fibers melting behavior that is beyond the scope of this article and will be further studied in our next work. In this study, therefore, the result from DSC is still used to select the proper processing temperature range. From Table I, it is known that the incipient melting temperature and the melting peak of constrained PA6 fibers are 222.7°C and 228.0°C, respectively. Based on these two data, the processing temperatures are set as 222, 224, 226, and 228°C.

It is evident from Figure 4 and Table I that the recrystallized phase after fully melting has a lower-melting peak, a wider melting range, and lesser degree of crystallinity than the constrained PA6 fibers. The reason of these differences is that the recrystallized phase after fully melting PA6 fibers lost the high-orientation of original fibers and transformed into a new crystal form with lesser order than the original fibers.

### Partially melting behavior of PA6 fibers

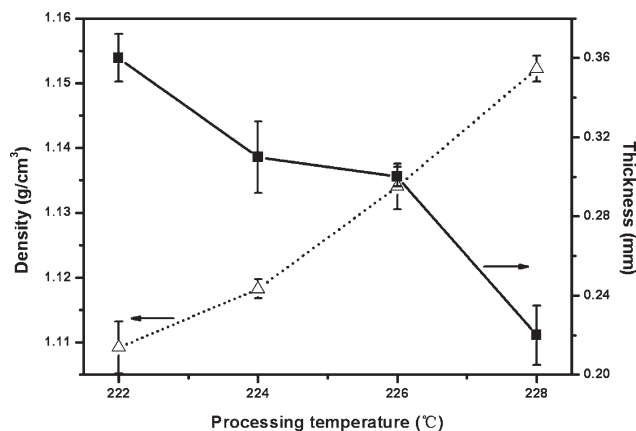
Making use of this remarkable difference of melting behaviors between the constrained PA6 fibers and the recrystallized PA6 phase after fully melting fibers, it is feasible to quantitatively investigate the effect of processing temperatures on the proportion of melting phase to original PA6 fibers by DSC.<sup>33</sup> In Figure 5, DSC melting endotherms of four  $\text{SPC}_{\text{PA6}}$  samples prepared at different temperatures are shown. For samples of  $\text{SPC}_{\text{PA6-222}}$ , only one melting peak appears during the course of melting by DSC. When the processing temperature is higher than 222°C, a lower-melting peak appears in the melting curves of  $\text{SPC}_{\text{PA6}}$  in addition to a higher melting peak and this lower-temperature peak



**Figure 5** Melting curves of SPC<sub>PA6</sub> prepared at four temperatures.

becomes more obvious with processing temperature increasing. At the same time, the position of higher melting peak moves to higher temperature and the size of it becomes smaller with processing temperatures increasing. Comparing with the two melting curves in Figure 4, it is not difficult to find that the higher melting peak of SPC<sub>PA6</sub> made at the temperatures higher than 222°C is a result of the remained PA6 fibers phase melting and the lower-melting peak is due to the melting of recrystallized PA6 phase after partially melting PA6 fibers.

Appearing only one melting peak during heating the SPC<sub>PA6-222</sub> sample by DSC proves that hardly any melting of PA6 fibers took place at the processing temperature of 222°C. During the process at a higher temperature than 222°C, surface of PA6 fibers began to melt owing to more free volume on the fiber surface than inside. When increasing processing temperatures, more part of PA6 fibers surface lost the original higher-orientation and recrystallized to form a lower-order phase after cooling. Therefore, this newly formed PA6 phase possesses a lower-



**Figure 6** Effect of processing temperatures on density and thickness of SPC<sub>PA6</sub> sheets.

**TABLE I**  
Part Performance Parameters of PA6 Fibers

Diameter (μm)	Molecular mass (×10 <sup>4</sup> )		Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)	Constrained fibers			Recrystallized phase after fully melting fibers						
	M <sub>n</sub>	M <sub>w</sub>				T <sub>onset</sub> (°C)	T <sub>peak</sub> (°C)	T <sub>end</sub> (°C)	ΔH <sub>c</sub> (J g <sup>-1</sup> )	X <sub>c</sub> (%)	T <sub>onset</sub> (°C)	T <sub>peak</sub> (°C)	T <sub>end</sub> (°C)	ΔH <sub>c</sub> (J g <sup>-1</sup> )	X <sub>c</sub> (%)
20	1.3	2.4	590	11	5.4	222.7	228.0	230.5	69.8	30.3	212.0	220.5	223.8	42.4	18.4

**TABLE II**  
Crystallization Parameters of SPC<sub>PA6</sub> Prepared at Different Processing Temperatures

Sample no.	Processing temperature (°C)	Fiber phase		Recrystallized phase after partially melting		X <sub>c</sub> of SPC <sub>PA6</sub> (%)
		T <sub>peak</sub> (°C)	ΔH <sub>c</sub> (J g <sup>-1</sup> )	T <sub>peak</sub> (°C)	ΔH <sub>c</sub> (J g <sup>-1</sup> )	
SPC <sub>PA6-222</sub>	222	226.2	67.5	–	–	29.3
SPC <sub>PA6-224</sub>	224	229.6	59.9	220.5	0.1	26.1
SPC <sub>PA6-226</sub>	226	231.3	46.5	222.3	3.3	21.7
SPC <sub>PA6-228</sub>	228	234.8	12.4	225.7	39.3	22.5

melting temperature than original PA6 fiber. The melting part of PA6 fibers flew to fill the gap between fibers under the applied pressure that resulted in the decrease of free volume around the remained PA6 fibers in the final SPC<sub>PA6</sub>. This reduce of voids can be proved by the increase of density of SPC<sub>PA6</sub> sheets with processing temperatures raising, as shown in Figure 6. The gradual decrease of thickness of SPC<sub>PA6</sub> sheets with increasing processing temperature was a result of filling the voids by melting part of PA6 fibers and some of the melting part flowing out the sheets under the pressure. The free volume around PA6 fibers reducing contributes to the shift of melting peak of the remained PA6 fibers in SPC<sub>PA6</sub> to higher temperature position and this change can be clearly observed in Figure 5.

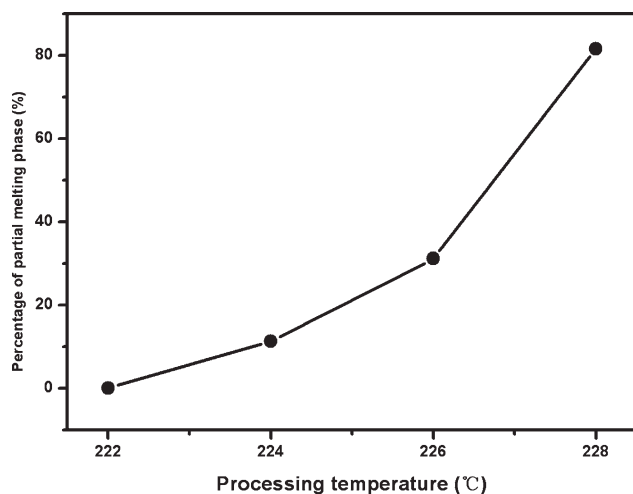
Crystallization parameters of four SPC<sub>PA6</sub> samples are listed in Table II. The ratio of partially melting PA6 phase to the original PA6 fiber is calculated by comparing the melting enthalpy of recrystallized phase after partially melting with that of original PA6 fiber (69.8 J/g, see Table I) and the results are shown in Figure 7. It is very clear that increasing processing temperature is in favor of melting more part of PA6 fibers. For sample SPC<sub>PA6-222</sub>, hardly any part of PA6 fibers were melted during the molding process. However, at the processing temperature of 228°C, 82% of

PA6 fibers lost the original high-orientation and formed a new crystal texture having a lower-melting-point after cooling. From Table II, it is also concluded that the overall crystallinity of SPC<sub>PA6</sub> reduced with the processing temperatures increasing. This decrease of crystallinity contributed to the transform of SPC<sub>PA6</sub> sheets from opaque to translucence with the raise of processing temperatures, as shown in Figure 2. The other reason of this transform in the transmission of light is that interface performance improved with processing temperatures increasing due to melting more part of PA6 fibers to bond the fibers together.

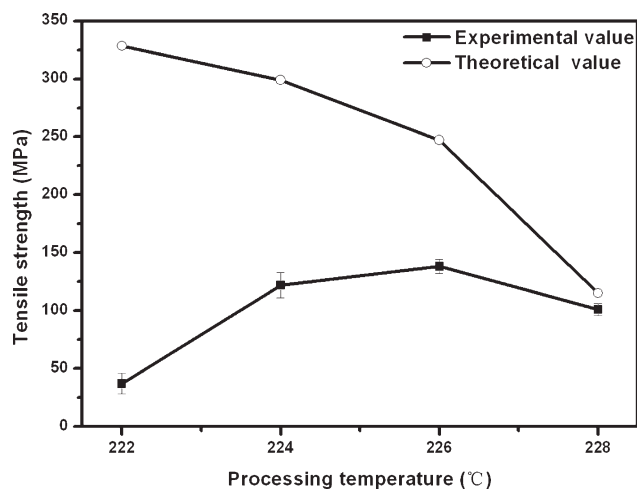
Molten part of PA6 fibers no longer possessed the high orientation of original PA6 fibers that would harm the strength of SPC<sub>PA6</sub> along the fiber direction. On the other hand, the molten part filled the voids in the original PA6 cloth and bonded the fibers together to form a complete consolidation structure of SPC<sub>PA6</sub> after recrystallization by cooling. Therefore, it is essential to determine an optimal percentage of partially melting phase to original PA6 fiber to achieve a first-class performance of SPC<sub>PA6</sub>.

### Performances of SPC<sub>PA6</sub>

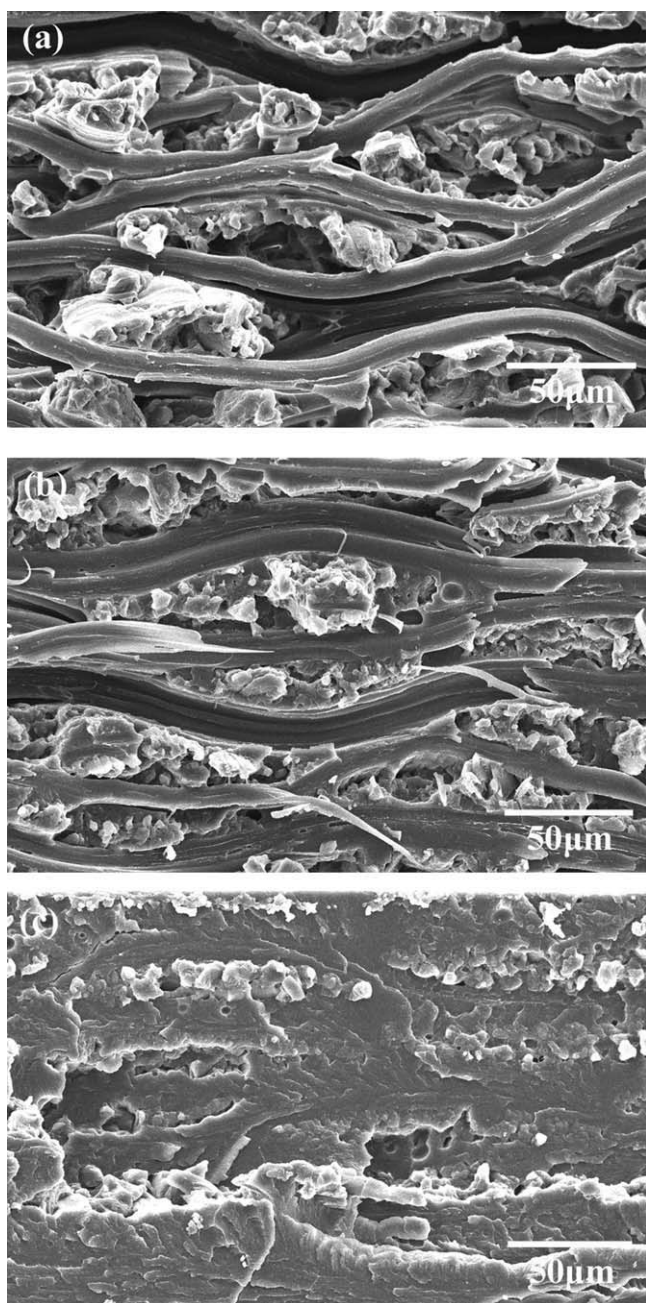
To obtain the optimal ratio of partially melting phase to original PA6 fibers, the tensile tests are



**Figure 7** Influence of processing temperatures on the percentage of partially melting phase to original PA6 fiber.



**Figure 8** Influence of processing temperatures on the tensile strength of SPC<sub>PA6</sub>.



**Figure 9** Cross-sectional morphologies of three samples: (a) SPC<sub>PA6-224</sub>, (b) SPC<sub>PA6-226</sub>, and (c) SPC<sub>PA6-228</sub> after tensile fracture by SEM.

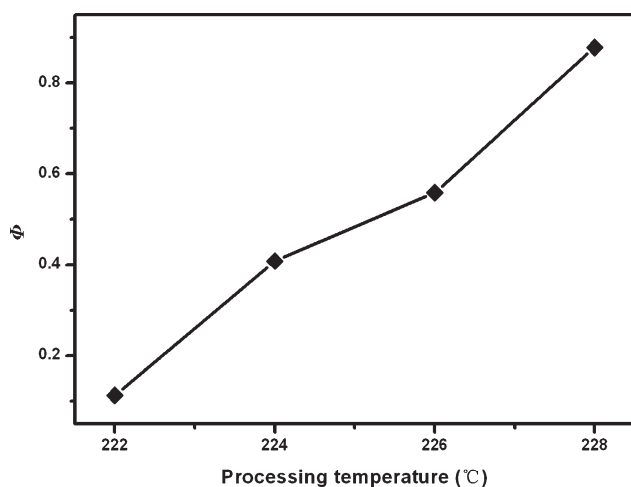
performed on four SPC<sub>PA6</sub> samples that were prepared at different temperatures. Change of tensile strength of SPC<sub>PA6</sub> with processing temperature is shown in Figure 8. As a result of no melting of PA6 fibers at 222°C that is proved by the single melting peak of SPC<sub>PA6-222</sub> in Figure 5, the sheet of SPC<sub>PA6-222</sub> was likely formed by the physical entanglement of PA6 fibers and effective interface bonding did not happen and original voids still existed inside. Therefore, the tensile strength of samples SPC<sub>PA6-222</sub> presented a lower value of 37MPa. With

processing temperatures increasing, partially melting of PA6 fibers took place and some voids were filled by molten PA6 phase and effective fiber/matrix interface bonding was formed in SPC<sub>PA6</sub> sheet, which could be proved by the uneven fiber surface in Figure 9(a). For this reason, the tensile strength of SPC<sub>PA6</sub> increased with the raise of processing temperatures and reached a maximal value of 138 MPa at 226°C. For SPC<sub>PA6-226</sub>, it is known from Figure 7 that the percentage of partially melting phase to original PA6 fibers is 31%. This value of about 30% conforms to the result reported in Ref. <sup>17</sup>. When processing temperature increased further to 228°C, a majority of PA6 fibers (82%, see Fig. 7) was melted and the orientation degree of PA6 molecules along fibers direction decreased severely. Even though, melting more part of PA6 fibers contributed to decreasing the void fraction that is proved by the increase of density of SPC<sub>PA6</sub> in Figure 6. Melting more part of PA6 fibers favored improving the interface bonding that is illustrated by the cross-sectional morphologies of SPC<sub>PA6</sub> after tensile fracture in Figure 9 (the cross-sectional morphology of SPC<sub>PA6-222</sub> is not shown here because of no complete fracture taking place after tensile failure): with increasing processing temperature, it was more difficult to distinguish the remained PA6 fibers in the cross section and the cross section became more even; this change of fracture morphologies indirectly inflected that the interface bonding improved with processing temperature increasing. Therefore, the tensile strength of SPC<sub>PA6-228</sub> had a lower value than that of SPC<sub>PA6-226</sub>.

According to a parallel rule of mixtures for the tensile strength of the plain weave reinforced composite,<sup>30</sup>

$$\delta_c = \delta_f \frac{V_f}{2} + \delta_m (1 - \frac{V_f}{2}) \quad (1)$$

where  $\delta_c$ ,  $\delta_f$ , and  $\delta_m$  are the tensile strength of the composites, fibers, and matrix, respectively, and  $V_f$  is the volume fraction of fibers, a theoretical value of composite tensile strength can be obtained. In this study, the densities of PA6 fiber and newly formed PA6 matrix by partially melting fibers are almost the same, so the volume fraction of fibers can be achieved from Figure 7. Taking the result from Table I gives  $\delta_f = 590$  MPa. For the PA6 matrix, the tensile strength of neat PA6 sheet prepared at 250°C is used as  $\delta_m$  (67 MPa). After calculation, a series of theoretical upper limit for the tensile strength of the SPC<sub>PA6</sub> ( $\delta_c$ ) can be obtained and are shown in Figure 8. By comparing this theoretical value with the experimental one ( $\delta_e$ ), a coefficient ( $\varphi = \delta_e/\delta_c$ )<sup>13</sup> inflecting the reinforcing effect of PA6 fibers on the PA6 matrix is obtained and shown in Figure 10. For the case of SPC<sub>PA6-222</sub>, a lower coefficient value of 0.11 is a result of that nearly no PA6 fibers was melted to



**Figure 10** Comparison of experimental tensile strength of  $SPC_{PA6}$  with theoretical ones.

form matrix (Fig. 7) and the complete consolidation structure of composites did not take shape. Increasing the processing temperature has a positive effect on this coefficient, which proves that melting more part of PA6 fibers was in favor of forming an integrated consolidation structure in  $SPC_{PA6}$ . It is mentionable that for  $SPC_{PA6-228}$ , the coefficient reached up to 0.88 reflecting the obvious reinforcing effect of PA6 fibers on PA6 matrix in this kind of composites.

## CONCLUSIONS

Single polymer composites were successfully fabricated by precisely controlling the partially melting behavior of PA6 fibers under the action of heat. Only the recycled PA6 fibers that were taken from ordinary costuming were used during the whole process. This new kind of material possesses a complete consolidation structure of composites and remarkable reinforcing effect of polymer fibers on polymer matrix. Its tensile strength reached a maximal value of 138 MPa at the processing temperature of 226°C. It is foreseeable that, due to the same thermoplastic polymer nature of fiber and matrix, this material can be postformed into complex parts only by thermal deformation and promises a better recyclability by fully melting than traditional glass or carbon fibers reinforced polymer composites.

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