Manufacturing and Properties of Nanofiber Composites Using a PAR/PET Sheath-core Fiber

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ABSTRACT

To manufacture a PAR(Polyarylate) nanofiber reinforced PET(Polyethylene terephthalate) composite, a sheath(PET)-core(PAR) fiber was spun by a conjugate spinning. Using the conjugated fiber, a PAR/PET composite can be obtained. The effects of the PAR content and the spinning speed on the properties of the conjugated fiber and its composite were investigated. Tensile strength and degree of crystallization were analyzed by the tensile tester and XRD(X-ray diffraction). Consquently, tensile strength and degree of crystallization increased as the increase of the PAR content and spinning speed of the conjugated fiber and its composite.

Keywords: PAR, LCP(Liquid crystal Polymer) fiber, PET, Sheath-core fiber, Nanofiber composite

1 INTRODUCTION

Recently, a fiber reinforced composite is in the spotlight as a specialized-functional material having high strength, modulus and elasticity. Due to these properties, fiber reinforced composites have been applied for various industrial fields such as aerospace and civil engineering etc. Especially, when the LCP nanofiber having high performance is used as a reinforcement in the composite, excellent properties compared to existing are expected. The PAR fiber is a typical LCP fiber and has high strength, modulus and thermal resistance.

In this study, PAR reinforced PET nanofiber composite was manufactured using a sheath-core fiber by conjugate spinning. The core phase in the conjugated fiber is PAR nanofiber and the sheath phase is PET which is a wide use polymer. We analyzed various properties of the conjugated fiber and PAR/PET nanofiber composite with the PAR content and spinning speed of the conjugated fiber.

2 EXPERIMENTALS

2.1 Preparation

The PAR, core phase, is prepared using Vectra® from TICONA in Japan and the PET resin was provided from HYOSUNG in Korea. To prepare the PAR/PET sheath-core fiber, PAR and PET resin were melt spun by a conjugated spinning with various conditions on the spinning speed and PAR content. The conditions of conjugate spinning are shown in Table 1 and the cross-sectional view of the conjugated fiber is shown in Figure 1.

Table 1. Experimental spinning conditions of sheath-core fiber

Condition	Variables
Spinning speed (mm/min)	300, 400, 500, 600 (Content of core : 50%)
Content of core (%)	30, 50, 70 (Spinning speed : 500m/min)

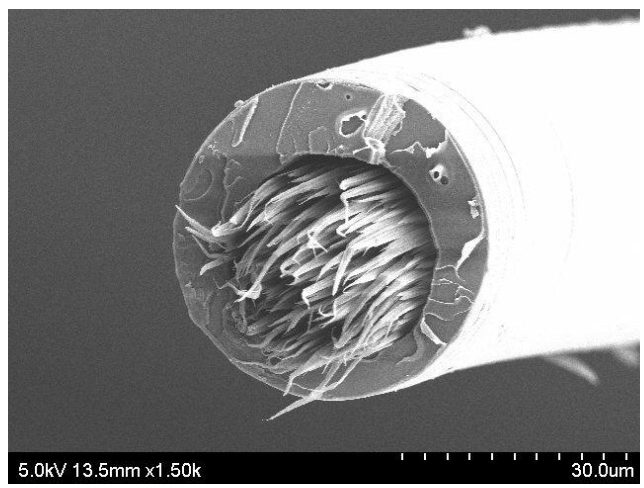

Figure 1. Cross-sectional view of the PAR/PET sheath-core fiber

2.2 Properties of conjugated fiber

Tensile strength

Tensile strength of the conjugated fiber was performed by a ASTM D 3822 using an Instron®4467. In the test, load cell and cross-head speed were fixed 50N and 1mm/min, respectively. To minimize the influence of moisture, conjugated fiber was dryed for 30minutes under 80°C before the evaluation.

X-ray diffraction

Degree of crystallization of the conjugated fiber was investigated with a XRD(X-ray diffractometer, D/MAX-2200 Ultima/PC of Rigaku International Co.). We used a Wide-Angle X-ray Scattering(WAXD, Pruker, D8 Discover) and CuK radiation(λ=1.5406Å).

2.3 Manufacturing of the PAR/PET nanofiber composite

To manufacture a PAR/PET nanofiber composite, conjugated fiber wound up uni-directionally in a steel plate and it was molded in a hot press for 10minutes under 265°C and 1000psi. Then, the PET sheath was melted and PAR nanofiber core maintained their shape in the composite. So, PET became a matrix and PAR became a reinforcement of the composite. Through this process, we can obtain a PAR nanofiber reinforced PET composite and schematic diagram of this process is shown in Figure 2.

Figure 2. Fabrication process of the PAR/PET nanofiber composite by using a sheath-core fiber

2.4 Properties of PAR/PET nanofiber composite

Tensile strength

Tensile test of the prepared PAR/PET nanofiber composite was performed by referring to ASTM D 638 using an Instron®4467. In the test, cross-head speed was fixed 1mm/min and sample size is shown in Figure 3.

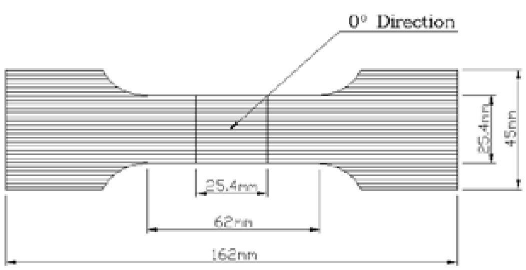

Figure 3. Schematic diagram of test sample

X-ray diffraction

Degree of crystallization of the PAR/PET nanofiber composites was analyzed using a XRD. X-ray exposure time was 300s at room temperature and we used a 2D-WAXD and CuK radiation(λ=1.5406Å).

3 RESULTS AND DISCUSSION

3.1 Properties of conjugated fiber

Tensile strength

In Figure 4, according to the spinning speed, tensile strength of the conjugated fiber increased with the spinning speed. It can be discussed with the spinning speed that the higher molecular orientation and more nanofibrous stucture might be obtained with the higher speed. These orientation along the fiber axis and development of the nanofibrous stucture contributed the increase of the fiber strength.

As the PAR content of conjugated fiber increases, tensile strength increases sharply as shown in Figure 5. Also, this phenomena can be explained with the nanofibrous stucture development of the PAR with the PAR core content.

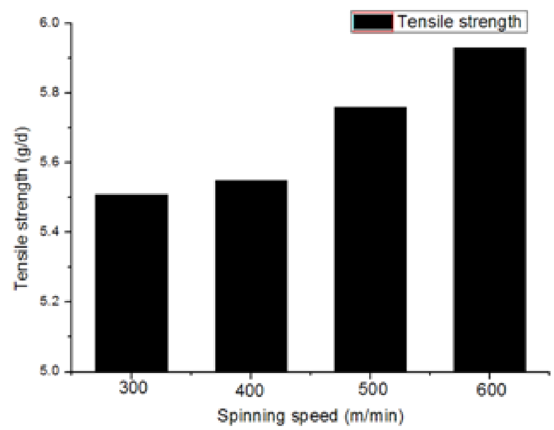

Figure 4. Tensile strength of the conjugated fiber as a spinning speed

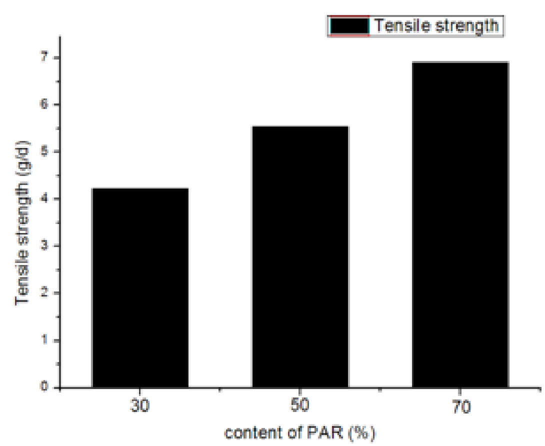

Figure 5. Tensile strength of the conjugated fiber as a content of PAR

Degree of crystallinity

As a result of performing XRD analysis, 1D-WAXD pattern was obtained. At the angle of 20° point, a peak of crystallization appeared in Figure 6. Also, it indicates that the degree of crystallization of conjugated fiber spun at 300m/min of spinning speed relatively low compared with other cases. The peak of crystallization at 400, 500 and 600m/min, on the other hand, are not significantly different. Figure 7 shows a XRD graph according to the PAR content and it indicates that as the content of PAR increases, peak of crystallization becomes higher and sharper.

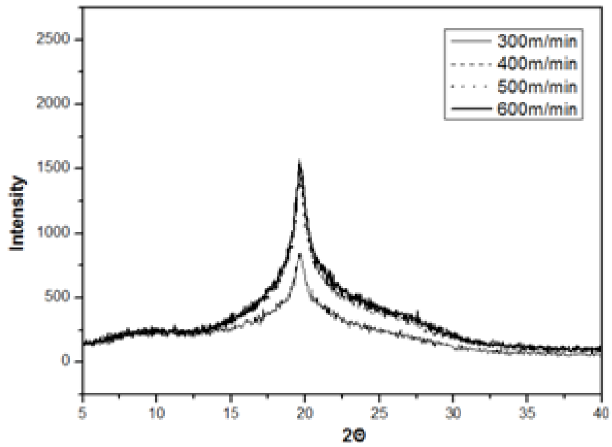

Figure 6. 1D-WAXS scan of the conjugated fiber as a spinning speed

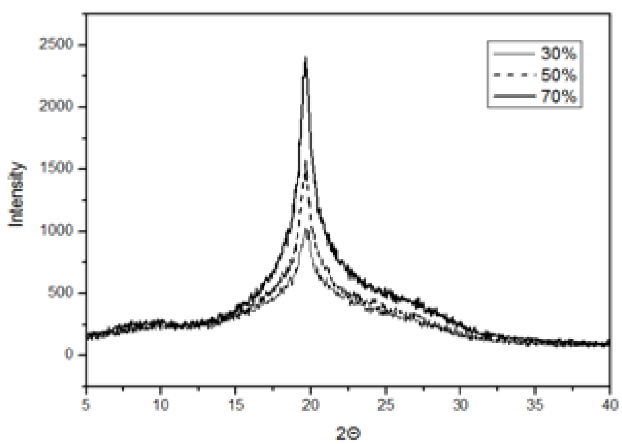

Figure 7. 1D-WAXS scan of the conjugated fiber as a content of PAR

3.2 Analysis of properties of PAR/PET nanofiber composite

Tensile strength

Tensile strength of the PAR/PET composite with spinning speed and PAR content are shown in Figure 8 and Figure 9. On the condition of spinning speed, tensile strength increases overall. Especially, difference tensile strength of nanofiber composite used spun 300m/min and 400m/min are not significantly. Whereas, these 500 and 600m/min are different significantly. In other word, tensile strength of nanofiber composite increase as more contain PAR in the conjugated fiber used manufacturing this composite.

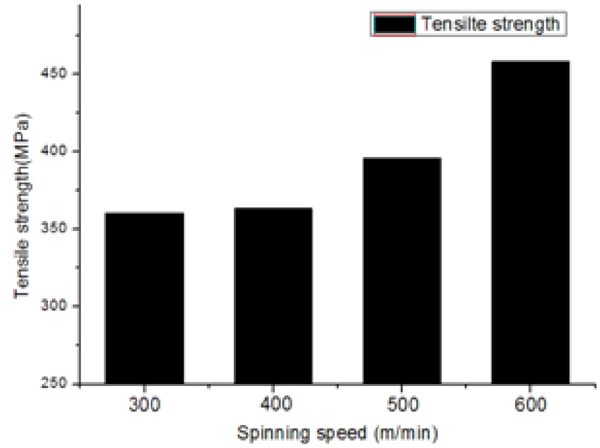

Figure 8. Tensile strength of the nanofibercomposite as a spinning speed on the conjugated fiber

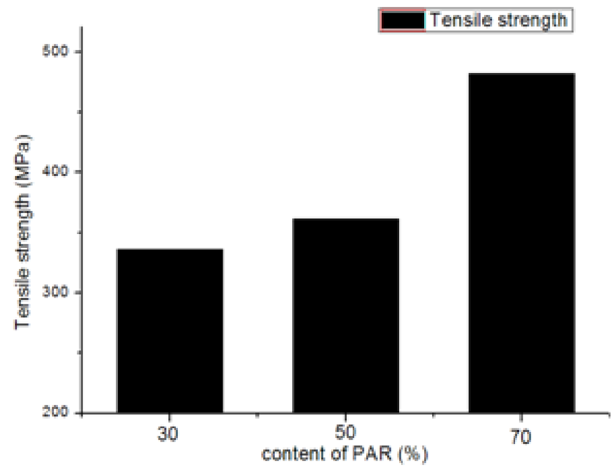

Figure 9. Tensile strength of the nanofibercomposite as a content of PAR on the conjugated fiber

X-ray diffraction

The area percentage of crystalline peak which is obtained with the XRD graph indicates a degree of crystallization. A relative crystallinity(%) value was calculated using an equation(1).

$$\text{Relative crystallinity(\%)}=\frac{A_c}{A_a+A_c}\times 100 \qquad (1)$$

Where, A_a is area of amorphous peak and A_c is area of crystalline peak. Figure 10 and 11 are results of XRD analysis of the PAR/PET nanofiber composite as spinning speed and PAR content.

Examining a crystalline structure of nanofiber composite, peaks of 010, 110, 100 plane, 2θ=14°,20°, 28° stem from the PAR's crystalline structure while 2θ=17°,22.5, 21° due to a similar pattern of the PET.

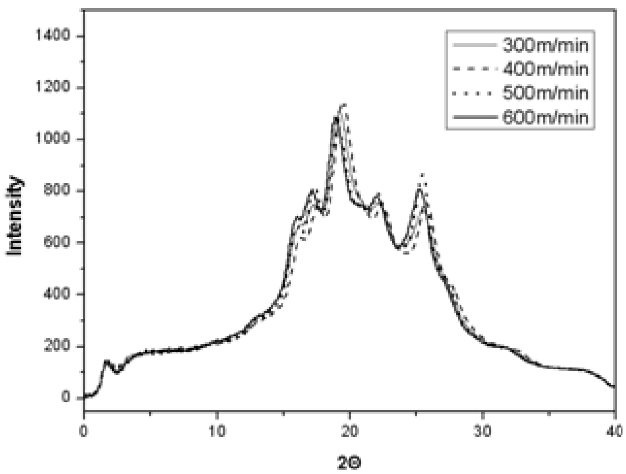

Figure 10. 1D-WAXS Scan of nanofibercomposite produced in accordance with melt spinning speed.

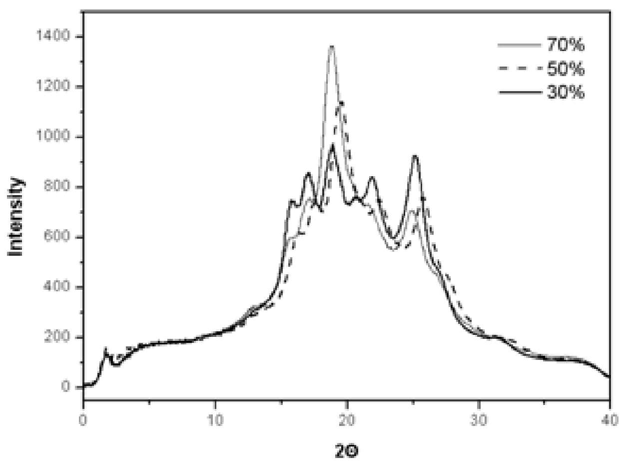

Figure 11. 1D-WAXS Scan of nanofibercomposite produced in accordance with content of PAR.

When the fiber spinning speed is up to 500m/min, degree of crystallization increases to some extent as shown in Figure 10. In Figure 11, the peak of XRD graph becomes sharper as the PAR content in the conjugated fiber.

4 CONCLUSIONS

In this study, the tensile strength of conjugated fiber and PAR/PET nanofiber composite increased as spinning speed and content of PAR of the sheath-core fiber increased. Because PAR core phase having high strength, tensile strength increase as a content of PAR in the conjugated fiber.

Crystallization and orientation behavior of conjugated fiber and nanofiber composite increased as spinning speed and content of PAR. It is caused that the PAR nanofibrous structure maintain its orientation in the conjugated fiber during the spinning. With the increase of the spinning speed, PAR nanofilaments in the conjugated fiber are oriented well. In addition, due to PAR which is a typical LCP fiber, orientation and crystallization increase as a content of PAR. Therefore, spinning speed and content of PAR are the main factors for determining the properties of the PAR/PET conjugated fiber and its nanofibrous composite.

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