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Effect of Heat-setting Temperature on the Structure and Performance of Ultra-fine Denier PET Full Drawing Yarn

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Abstract. The ultra-fine denier PET full drawing yarn (PET-FDY) with monofilament linear density equal to 0.38dTex was produced. During the practical process of ultrafine denier PET-FDY, the yarn should be heat-set at certain temperature following the drawing process in order to improve the mechanical properties and reduce the boiling water shrinkage. The influence of heat-setting temperature on the structure and performance of ultra-fine denier PET FDY was studied in this paper. With heat-setting temperature increasing, crystalline in PET FDY became perfect and crystallite size became bigger. According to the density method, crystallinity was augmented. Crystal orientation factor and overall orientation factor were both increased. The tenacity and tensile stress at 10% strain of PET FDY were increased and the breakage percentage and boiling water shrinkage were reduced with the heat-setting temperature increasing.

Introduction

Poly (ethylene terephthalate) fiber (PET) has many advantages, such as high strength and modulus, low hygroscopicity, good dimensional stability, so it is used widely in many fields[1]. In order to extend its application fields, differential PET fibers were developed in recent years, such as ultra-fine denier FDY, profiled fiber, composite fiber and so on[2~4]. Because of the very low monofilament linear density of ultra-fine denier fiber, the production process parameters, such as heat-setting temperature, draw ratio, velocity of wind and so on, influence the structure and performance of such fiber greatly. In this paper, the effect of heat-setting temperature on the structure of ultra-fine denier PET FDY was studied. During the practical process, FDY is drawn under different ratio, and then it is heat-set on the hot roller surface under certain temperature. In the heat-setting process, the length of fiber maintain unvaried.

Experimental

PET FDY (RK617) with round section cross was supplied by Hengli chemical fiber Ltd. The specification of RK617 is 73dTex/192f and monofilament linear density is equal to 0.38dTex. The production process parameters were as followed: takeup velocity: 3765m/min, drawing ratio: 1.38. Heat-setting temperatures were chosen as 100^{o}C, 110^{o}C, 120^{o}C, 130^{o}C, 140^{o}C, 150^{o}C and labeled as T1, T2, T3, T4, T5 and T6 respectively.

The determination of crystal structure and crystal orientation was performed using X' pert-pro MPD (PANalytical Netherlands) diffractometer. Radial scans of intensity versus scattering angle (2θ) were recorded in range 10~50° with identical setting of the instrument by using filtered CuKα radiation, an operation voltage of 40 kV and a filament current of 40 mA. The XRD intensity curves for equatorial, meridional and azimuthal scans were measured using the same system. The crystalline orientation was determined from azimuthal intensity measurements on the reflection of ($\bar{1}$05) plane.

Crystallite size was calculated according to the Sherrer equation (Eq.1).

$$L_{hkl}(\overset{\circ}{A}) = K\lambda / B\cos\theta . \quad (1)$$

Where L_{hkl} is the mean dimension of crystallites perpendicular to planes *hkl*, K is the shape factor (0.9), λ is the wavelength of X-ray, 2θ is the Bragg's angle, B is the integral half-width of experimental profile (rad).

The overall orientation of PET FDY was measured using sonic velocity method and calculated according to the Eq.2 :

$$f_s = 1 - \frac{C_u^2}{C^2} \quad (2)$$

Where f_s is sonic velocity orientation factor of fiber, C_u is sonic velocity of the unoriented PET fiber (1.35 km/s), C is the sonic velocity of the fiber under investigation.

The density of PET FDY was measured on density gradient column using a mixture of carbon tetrachloride and n-heptane at 23℃. The crystallinity (X) of PET FDY was calculated from the density measurement of the fibers (ρ) by Eq.3:

$$X(\%) = \frac{\rho_c(\rho - \rho_a)}{\rho(\rho_c - \rho_a)} \times 100\% \quad (3)$$

The crystalline density (ρ_c) used was 1.455g/cm^3 and the amorphous density (ρ_a) used was 1.335g/cm^3.

Boiling water shrinkage (BWS) was measured under 100℃ water bath maintaining 30min. BWS was calculated by the Eq.4:

$$BWS(\%) = \frac{(L_0 - L_1)}{L_0} \times 100\% \quad (4)$$

L_0 was original length and L_1 was length after boiling.

All fibers were subjected to uniaxial strain in an Instron Testing machine (Model 3365). The measurements were carried out at room temperature and at a rate of 10mm/min.

Result and discussion

The equatorial diffraction profile and the meridional diffraction profile of different ultra-fine denier PET-FDY at different heat-setting temperature were shown in Fig.1 and Fig.2. From the Fig.1 and Fig.2, the XRD patterns of each PET-FDY clearly indicated that crystalline structure formed in the FDY. It can be seen that there are three main equatorial reflection and a main meridional reflection, corresponding to (010), ($\bar{1}$10), (110) and ($\bar{1}$05) planes respectively.

Intensity of related diffraction peaks was found to intensify and the FWHM was narrowed. As shown in the Table 1, the crystallinity and size of crystallite were both increased with the heat-setting temperature increasing, implying that the higher thermal energy (higher heat-treatment temperature) could lead more fractions of chains to crystallize [5]. We also found that under cold crystallization temperature (T_{cc}=135^{o}C), the crystallinity of PET-FDY increased. Because the length of fiber was constraint, Stress appeared to compensate the chain relax and then decrease of stress are evidently associated with extended-chain crystallization with temperature increasing.

From the Table 1, it can be concluded that the crystal orientation factor was increasing with the heat-setting temperature increasing. The sonic velocity orientation factor of ultra-fine PET FDY at different heat-setting temperature was shown in the Fig.3. Sonic velocity orientation factor is

composed of crystal orientation and amorphous orientation which indicated orientation of the whole molecular chain because of the long wavelength. From the Fig. 3, it can be concluded that the whole orientation factor was increasing with the heat-setting temperature increasing. During the heat-setting process of PET-FDY, because the length of fiber was held constant, the force appeared in the fiber in order to compensate the thermal shrinkage caused by the entropic recovery of oriented chains. So the crystalline orientation was enhanced with the heat-setting temperature increasing.

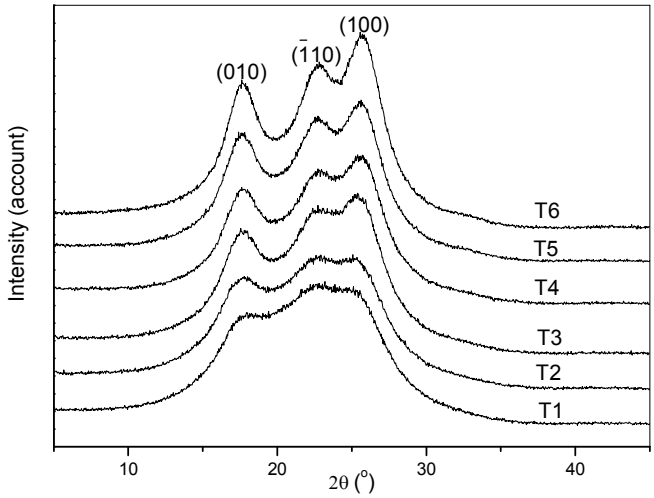

Fig. 1 Equatorial diffraction profile of PET FDY at different heat-setting temperature

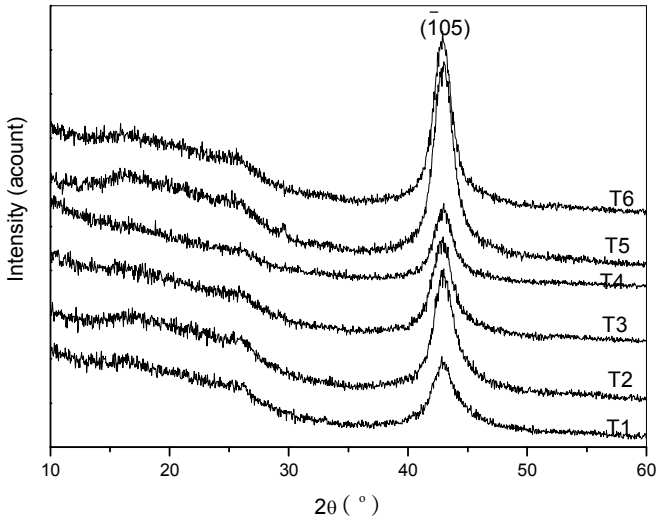

Fig.2 Meridioanl diffraction profiles of PET FDY at different heat-setting temperature

The improvement of crystallinity and orientation of ultra-fine PET FDY influence the performance and process of FDY greatly. The breakage stress and tensile stress at 10% strain of different fiber were shown in Fig.4. The heat-setting temperature has little influence on the breakage stress, but has greatly influence on the tensile stress at 10% strain. It means that PET FDY heat-setting at higher temperature will has better modulus because of the higher crystallinity and overall orientation. Tensile stress at 10% strain tended to constant, when the heat-setting temperature was higher than 140^{o}C The higher stress means that ultra-fine denier PET-FDY has good anti-deformation ability.

Table 1 Structure of ultra-fine PET FDY at different heat-setting temperature

	Crystallite dimension [Å]					
sample	$\bar{1}05$	010	$\bar{1}10$	100	x%*	f_c
T1	36.6	29.1	27.5	29.4	18.3	0.889
T2	44.5	36.2	39.0	31.5	22.2	0.925
T3	44.9	39.9	40.9	32.6	26.7	0.932
T4	48.7	40.6	41.8	34.4	28.8	0.944
T5	49.2	42.0	44.2	35.0	31.9	0.959
T6	51.6	45.7	44.6	35.5	35.7	0.979

* Measured by density method.

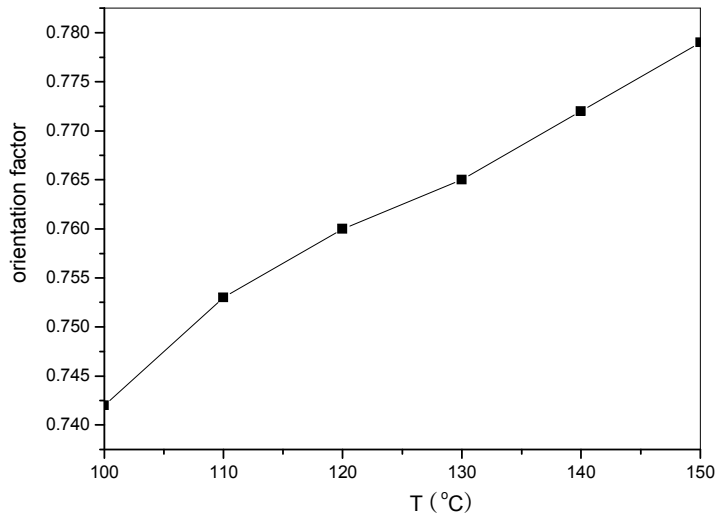

Fig.3 sonic velocity orientation factor of PET FDY at different heat-setting temperature

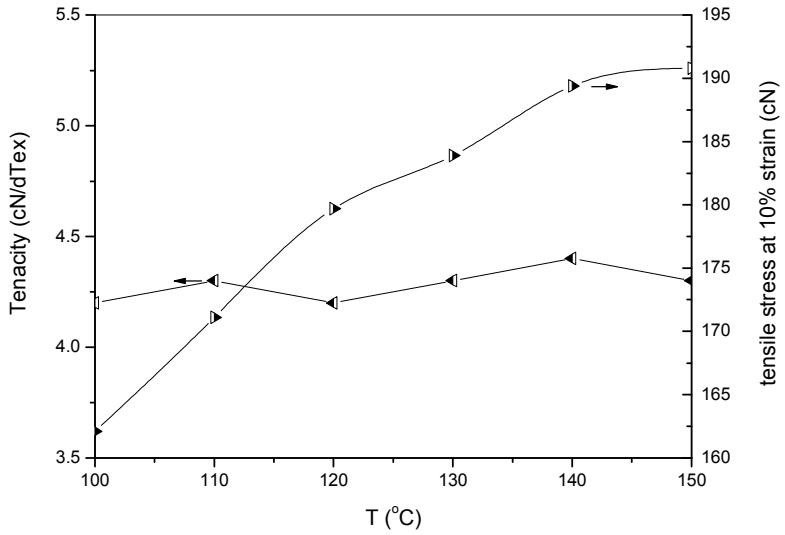

Fig.4 Tenacity and tensile stress at 10% strain of different PET FDY

The breakage percentage and boiling shrinkage of PET FDY heat-setting at different temperature are shown in Fig.5. Although the breakage percentage and boiling shrinkage both reduced with heat-setting temperature increasing, the tendency is different with each other. The breakage percentage reduced with the heat-setting temperature increasing and tended to constant when temperature reached 140°C.

From BWS curve in the Fig.5, it was interesting that there were two platform and two rapid reducing area. The ultra-fine deinier PET-FDY was drawn under certain draw ratio and then orientation took place in the fiber. When the heat-setting temperature was lower, the crystallinity was lower and BWS was higher. With the heat-setting temperature increasing, the crystallinity increased. Crystal domain can be regard as physical crosslinking area and limited the BWS. There were competition role of shrinkage and elongation and elongation is dominant when temperature was higher than 140^{o}C, So the amorphous orientation become lower and the crystallinity was relative high which caused BWS reduced[6, 7].

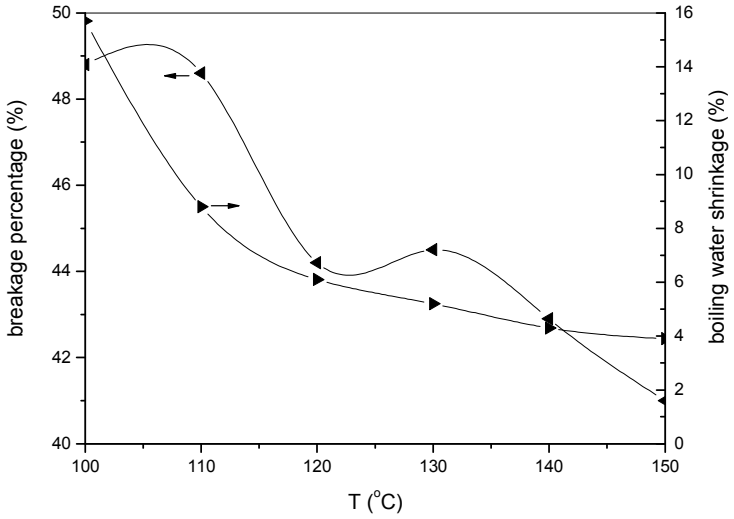

Fig.5 Breakage percentage and boiling water shrikage of different PET FDY

Conclusion

During the practical process of ultra-fine denier PET-FDY, fiber was heat-set following the drawing process and the length was regarded constant. The heat-set temperature had great influence on structure and performance of ultra-fine denier PET-FDY.

With increasing the heat-setting temperature, the perfection and size of crystallite and crystallinity were improved and the crystalline orientation and overall orientation were increased.

During the testing condition, the heat-setting temperature had little influence on the tenacity, but had great influence on the tensile stress at 10% strain, breakage and BWS.

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