Comparison of Tensile, Thermal, and Thermo-Mechanical Properties of Polyester Filaments Having Different Cross-Sectional Shape

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Received 6 June 2008; accepted 27 July 2008
DOI 10.1002/app.29097
Published online 13 October 2008 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: One of the most important morphological features of fibers is their cross-sectional shape. Nowadays, the circular fiber cross-section is the most common shape of melt-spun man-made fibers. Other shapes are beginning to emerge for a variety of reasons such as performance, comfort, pilling propensity, bulkiness, tactility, processing etc. The filaments' cross-section can be easily varied by changing the spinneret hole shape. Synthetic fibers that are predominantly spun by the melt spinning method with spinnerets having the noncircular hole geometry are called profiled or noncircular fibers. Modifications of the fiber cross-section allow designing surface properties in yarn and fabric. However, the effect of profiled fibers on yarn properties has not been well documented yet. In this article, the influence of different filament cross-section geometry on fiber properties was studied. PET (polyethylene terephthalate) filament yarns having two different cross-sectional shaped filaments, circular and cruciform, were manufactured by melt spinning. Differences in tensile properties of filament yarn and as well as of individual filament depending on the cross-sectional type were studied and revealed. More over, thermal and thermomechanical properties of filament yarn of both the cross-sections were studied and revealed by DSC and TMA method, respectively. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 111: 805–812, 2009

Key words: polyesters; profiled fibers; mechanical properties; thermal properties

INTRODUCTION

One of the most important morphological features of fibers is their cross-sectional shape. Nowadays, the circular fiber cross-section is the common shape manufactured by synthetic fiber producers. Other shapes are beginning to emerge for a variety of reasons such as performance, comfort, pilling propensity, bulkiness, tactility, processing etc.[1] The cross-section of a synthetic fiber produced by the melt-spinning method can be easily varied by changing the spinneret hole shape. Modifications of fiber cross-section allow to design surface properties in yarn and fabric.[2]

Filament cross-sections for the use in textiles and composites are becoming more and more complex. The main morphological feature of a melt-spun filament is its cross-sectional structure,[3] which can be divided into two general classes: (i) cross-sectional area (full or solid and hollow or tubular) and (ii) cross-sectional shape (circular and profiled or noncircular).

Synthetic fibers that are predominantly spun by the melt spinning method with spinnerets having the noncircular hole geometry are called profiled or noncircular fibers. The development of melt-spun fibers with profiled cross-sections started in the 1960s. The first attempt was made to mimic the gloss of expensive silk fibers by changing the cross-section to a triangular shape.[4,5] Since then, various types of noncircular fibers have been developed with the aim to add functionalities and esthetics to synthetic fibers leading to a change of their surface properties. The cross-section of a synthetic fiber produced by the melt-spinning method can be easily varied by changing the spinneret hole shape. Their orifices are not circular, as in a standard spinneret, but oblong, star shaped, or triangular.

In general, fibers consisting of noncircular cross-sectional shaped filaments show properties different from those of fibers with circular cross-sectioned ones, including the bending stiffness, coefficient of friction, softness, luster, comfort, pilling, bulkiness, handle, and performance.[5–7]

Poly(ethylenterephthalate) (PET) filament yarns made from two different cross-sectional shaped filaments, circular and cruciform, were manufactured

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Journal of Applied Polymer Science, Vol. 111, 805–812 (2009)

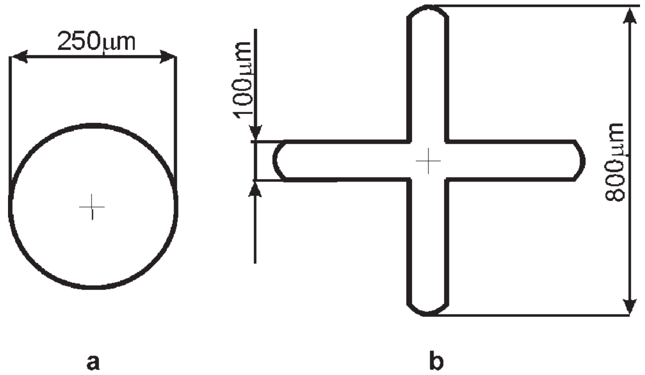

Figure 1 Sketch of spinneret capillary hole (a) circular; (b) cruciform.

by the melt spinning process. This article reports the influence of different filament cross-sectional geometry on fiber properties. The effects of the change in the fiber cross-sectional shape on tensile, thermal, and thermomechanical properties of PET filament yarn were studied. This work also includes the study of online measurement of the fiber velocity along the spin line.

EXPERIMENTAL

Melt spinning of circular and noncircular polyester filament yarn

Polyester filament yarns with two different cross-sectional shapes of filaments—circular and cruciform—were manufactured in the melt-spinning process by changing the spinneret having different cross-sectional shapes of capillary holes. Other spinning parameters were kept constant. The spinning speed was set to 3500 m/min.

Sketches of the spinnerets used are shown in Figure 1. As filaments emerged, they were drawn away from the outlet, stretching the polymer before it cools. Immediately after emerging, cool air was passed to solidify the melt. The solidified melt as a bundle of filaments came out and passed through a spin finish applicator by metering finish system. After that, the filament was wound onto a bobbin by a winder. No godet was used to draw the filaments produced. Thus, PET polymers converted from polymer chip into filaments by this process called the spin winding process. Melt temperature of 294°C, winding speed of 3500 m/min, number of filaments of 12, pressure before spin pump and after it of 50 and 80 bars, respectively, were chosen as technological parameters.

A noncontact measurement method[8] was used for online measurements of the fiber speed. The speed of moving filament at various distances from the spinneret with an interval of 10 cm was measured with a Laser Doppler velocimetry (LDV) device LaserSpeed LS50*M* (TSI, USA). The distance between the device and fibers was $\sim$ 280 mm, which can be adjusted in such way that two laser beams meet each other exactly at the fiber surface. The laser device was mounted onto a vertical stand and can be moved up and down along the spin line.

Fiber characterization

Optical microscopy was used to determine area and perimeter of circular and cruciform filaments. For these tests, the filaments were embedded in epoxy resin matrix followed by curing at ambient temperature. The specimens were polished perpendicularly to the fiber axis with a SiO_2/Al_2O_3 suspension down to an average grain size of 0.06 μm. After having cleaned in water and dried, the polished samples were analyzed using an optical microscope. The images of filament cross-sections, illustrated in Figure 2, were manually analyzed using an interactive computer graphic instrumentation IMAGEJ, calculating length, area, and pixel value statistics of user-defined selections.

From simple geometrical considerations, both the diameter of circular filaments and the equivalent diameter of cruciform filaments were estimated from

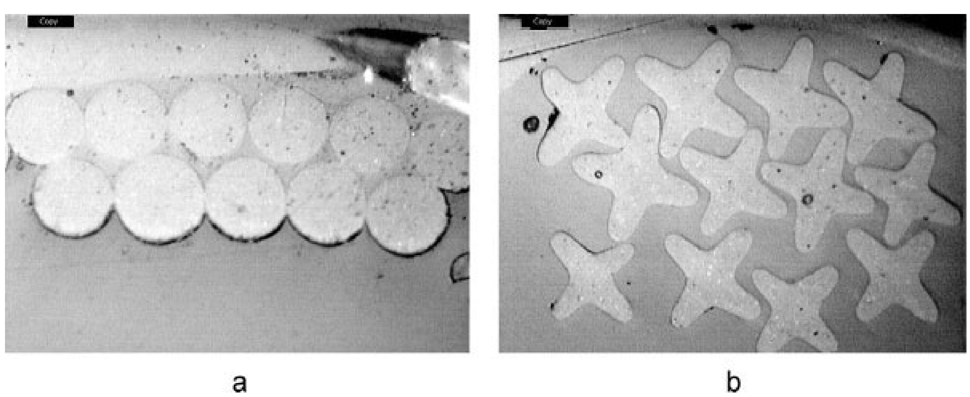

Figure 2 Microscopic images (cross-sectional view) of circular (a) and cruciform (b) cross-sectional shaped filaments.

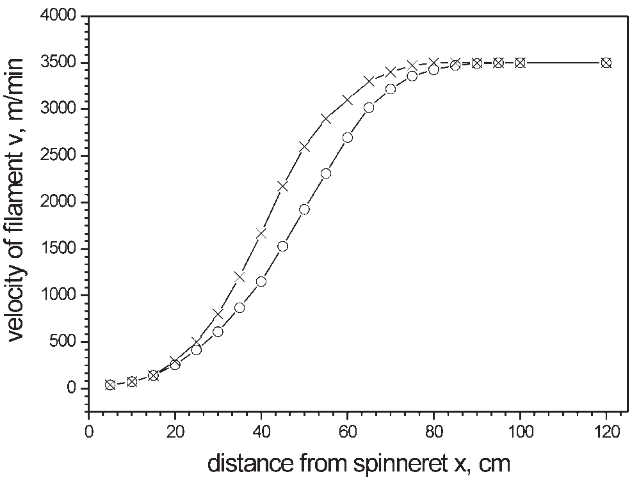

Figure 3 Velocity profile of circular (○) and cross-sectional (X) shape of PET filament yarn.

their area. The equivalent diameter of a noncircular filament is equal to the diameter of a circular filament having the same fineness. The values of area and perimeter were used to calculate the circularity ratio. The circularity ratio is a common compactness measure defined as the ratio of a shape area to the area of a circle (the most compact shape) having the same perimeter. This ratio is expressed as

$$M = \frac{4\pi A}{P_1^2} \tag{1}$$

where A and P_1 are the area and perimeter, respectively. The circularity ratio is equal to 1, $\pi/4$, and 0 for a circle, a square, and an infinitely long narrow shape, respectively.

Tensile test

The single fiber tensile measurement was conducted for at least 50 single fibers under stable conditions of temperature (23°C) and humidity (50%) using the Favigraph testing device (Textechno Herbert Stein, Mönchengladbach, Germany) equipped with a 100 cN force cell. The cross head velocity is 10 mm/min and the gauge length is 20 mm in accordance with EN ISO 5079. The fineness of each selected filament was determined by using the vibroscope method in accordance with ASTM D 1577.

To verify the effect of surface properties on the statistical distribution of fiber tensile strength, the cumulative fracture probability P was fitted by bimodal Weibull distribution through the least squares method, and the Weibull moduli m_1 and m_2 were calculated. Basically, the well known single two-parameter Weibull model[9] assumes that the strength of brittle materials is controlled by homogeneously distributed flaws with a single population. In reality, this is inappropriate because the classification of fracture can be based on two modes: (i) intrinsic failure characterized by fiber internal defects and surface defects; (ii) extrinsic failure characterized by surface flaws which are strongly surface property dependent. Therefore, a statistical bimodal is applied for fitting competing distributions. The cumulative probabilities of failure for the single and bimodal Weibull distribution function are given by following analytical equations[9], respectively:

$$\text{Single modal: } P = 1 - \exp\left[-\left(\frac{\sigma}{s_0}\right)^{m_0}\right] \tag{2}$$

$$\text{Bimodal: } P = f_i \cdot P_i + (1 - f_i) \cdot P_e \tag{3}$$

where P is the cumulative probability of failure ($i/(n+1)$) at the tensile stress σ. The parameters m_0 and s_0 are the Weibull shape factor (or modulus, slope of the distribution) and the scale factor (or characteristic strength) of fractured fibers, respectively. According to the range of our experimental results, the Weibull shape factor is representative of the separation, i.e., the homogeneity of the defects, whereas the scale factor is related to the severity of the distribution. f_i, P_i, and P_e are mixing parameter,

TABLE I
Comparison of Area, Perimeter, Circularity Ratio, and Diameter Between Circular and Cruciform Cross-Sectional Shaped Filaments

	Cross-sectional shape of filament					
	Circular			Cruciform		
Parameters of filament	Mean	σ	CV	Mean	σ	CV
Surface area (μm^2)	295.7	48.3	16.3	324.3	41.6	12.8
Perimeter (μm)	60.7	4.9	8.2	97.1	6.1	6.3
Circularity ratio, dimensionless	0.99	0.002	0.2	0.43	0.03	6.3
Diameter (μm)	19.4	1.6	8.2	–	–	–
Equivalent calculated diameter (μm)	–	–	–	20.3	–	–

σ is the standard deviation.
CV is the coefficient of variation $CV = \frac{\sigma}{\text{Mean}} \cdot 100\%$.

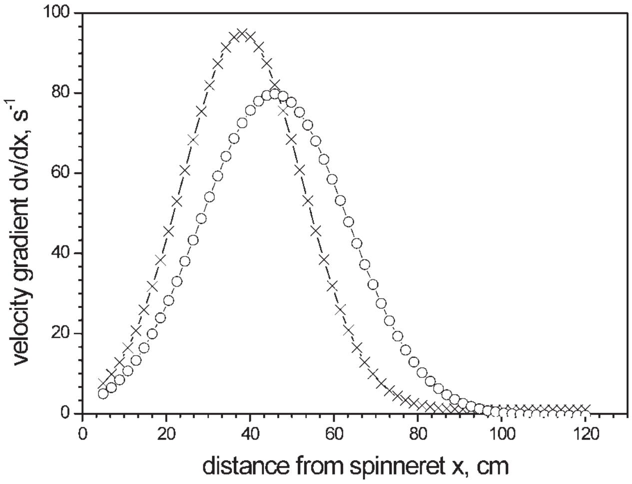

Figure 4 Velocity gradient profile of circular (○) and cruciform cross-sectional (X) shape of PET filament yarn.

probability density of intrinsic failure, and probability that a layer of thickness survives at a stress σ, respectively.

Yarn tensile tests were carried out according to DIN 53834 using a tensile strength testing device Goodbrand Micro 350 (Karl Schröder KG, Germany) with accuracy of about ±0.01 *N*. Samples of 100 mm yarn length were used and the yarn fineness determined by direct weighing was taken as an input parameter. The testing velocity was set to 200 mm/min. The tensile force versus deformation was recorded, and 10 measurements were taken to get the force average value for each type of yarn. Breaking force is a measure of the steady force necessary to break a fiber and is given experimentally by the maximum load developed in a tensile test.

Measurement of crystallinity degree using DSC

The crystallinity degree of PET filament yarns manufactured was studied by DSC using a Q1000 DSC instrument (TA Instruments, USA).

The following steps were carried out: (i) first heating from −60 to 300°C to obtain cold crystallization; (ii) cooling down from 300°C to −60°C and (iii) second heating from −60 to 300°C to find the maximum crystallinity.

The scanning rate and time period were used ±10 K/min (modulated) and 20 s, respectively. Samples of 5.006 mg of yarn with circular filaments and 4.993 mg of cruciform filaments were used for DSC measurements.

Thermomechanical analysis

The thermomechanical analysis (TMA) was carried out using a TMA Q400 instrument (TA Instruments, USA). The following modes were applied to characterize the thermomechanical properties of PET filament yarns manufactured: (i) standard mode, in which displacement is monitored at a constant force of 0.05*N*, under temperature increasing linearly with time from 20 to 200°C, at a heating rate of 3 K/min; (ii) modulated TMA mode, in which temperature is programmed linearly and simultaneously modulated at constant force (0.05*N*) to generate signals relating to total displacement and its reversing and nonreversing components. It is a way of separating the thermally reversing nature of linear expansion from irreversible changes in dimensions that accompany creep under load or relaxation of imposed stresses, such as orientation during manufacture. The heating rate was set to 3 K/min with a 300 s period, 3°C amplitude sinusoidal temperature modulation. Measurements were carried out in the temperature range from 25 to 200°C; and (iii) dynamic TMA mode, in which a sinusoidal stress within the interval from −0.1*N* to 0.1*N* at a frequency of 0.5 Hz was applied during a linear temperature ramp in the range from 30 to 150°C, at a heating rate of 3 K/min. The resulting data were used to calculate the material viscoelastic properties E', E'', and tan δ.

TABLE II
Fineness of PET Yarn and Filament of Circular and Cruciform Cross-Section Obtained by Direct Weighing Method

Filament cross-section	Yarn fineness (dtex)	Filament fineness (dtex)
Circular	55.0	4.58
Cruciform	56.3	4.69

RESULTS AND DISCUSSION

An idea about the fiber solidification point can be found from the fiber speed measurement results

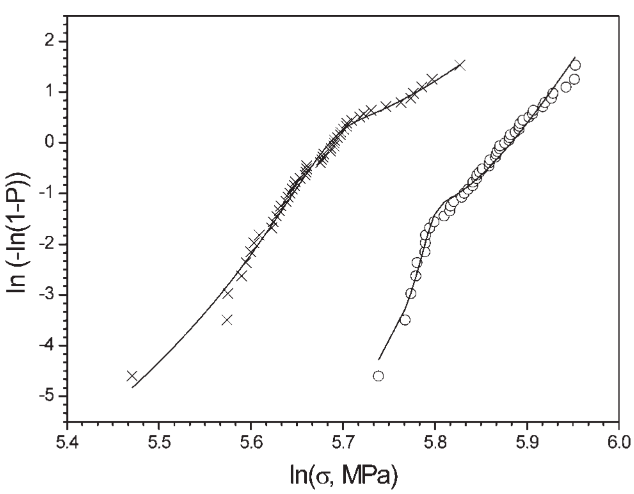

Figure 5 Single fiber tensile test data by bimodal Weibull CDF.

TABLE III
Tensile Properties PET Yarn made of Circular and Cruciform Cross Sectional Shaped Filaments

Filament cross-section	Properties	Mean	σ	CV
Circular	Breaking force (N)	1.33	0.05	3.49
	Tenacity (cN/tex)	24.33	0.85	3.49
	Elongation at break (%)	118.91	6.08	5.11
Cruciform	Breaking force (N)	1.03	0.06	6.14
	Tenacity (cN/tex)	18.36	1.13	6.14
	Elongation at break (%)	87.61	5.75	6.56

along the spin line. The solidification point is the region where the fiber becomes solid, i.e., the end of the fiber formation and this is the first point along the spinning line where the fiber reaches the take-up speed. Figure 3 shows the velocity profile of circular and cruciform cross-sectional shape of filament yarns along their spin line. It is clearly seen that the solidification point of filaments with cruciform cross-sectional shape shifted to a region close to the spinneret. This result indicates that cooling of filaments along the spin line was enhanced by the change of the cross-sectional shape from circular to cruciform. The enhancement of cooling for cruciform filaments is attributable to their larger surface area (about 60%; cf. Table I) per unit length compared to that of circular filaments.

Figure 4 shows velocity gradient profiles of filaments along the spin line, i.e., the rate of velocity change with respect to the change in distance from the spinneret. It shows that the maximum point of velocity gradient for cruciform filaments is higher than that for circular filaments and shifted toward the spinneret. Although the difference in solidification point of circular and cruciform filaments is not significantly high, this slight change contributes to a difference in their tensile properties, which will be discussed later.

The filament and yarn fineness obtained by direct weighing method are given in Table II. The fineness values for circular filaments slightly deviate from those for cruciform filaments. The reason for that may be deviations of the mass throughput. Spin finish used during melt spinning could also contribute to the slight deviation of the fineness. Moreover, deviations in measuring the exact test length of filament yarns before weighing them could also be a reason for the deviation in yarn fineness.

Area, perimeter, and circularity ratio of circular and cruciform cross-sectional shaped filament were obtained by optical microscopy. Values for area and perimeter obtained by optical microscopy are shown in Table I. Each mean value reported is an average value of ~ 30 measurements of each type of cross-section. The diameter of circular cross-section and the equivalent diameter for cruciform cross-sectional shape were calculated from the value of their areas, respectively. It can be seen that the perimeter of cruciform filaments increased by about 60% compared to that of circular cross-sectional shaped filaments for the same spinning speed and fineness. The higher surface area per unit length of cruciform filaments leads to more rapid cooling and the shift of their solidification point toward the spinneret during spinning.

The surface area values measured for the same shape type were not exactly equal to each other, as they should have been. The values of their standard deviations describe their difference in size. The reason for the deviations may be either because of the deviation in filament fineness arising from melt spinning or subjective errors during selecting the area from the microscopic images.

A detailed description of the single fiber tensile test data by bimodal Weibull CDF is shown in Figure 5. From the Weibull best-fit lines, the cruciform cross-section shaped filaments result in apparently lower Weibull plot lines and Weibull moduli ($m_1 = 15$, $m_2 = 33$). Interestingly, the lines for circular

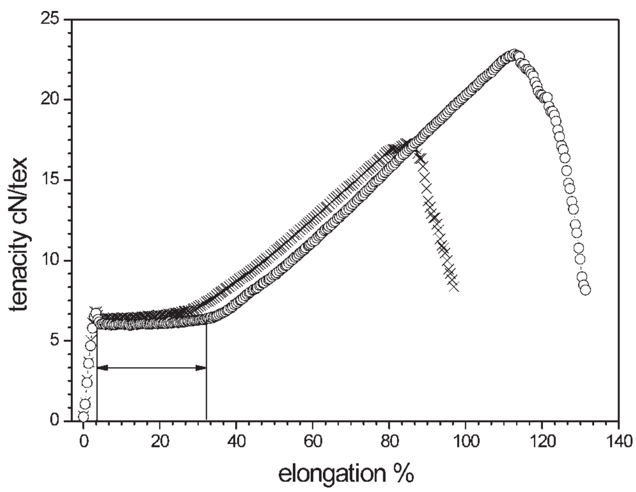

Figure 6 Average tenacity-elongation curves of 10 measurements of PET filament yarns manufactured from circular (○) and cruciform (X) shape of PET filament yarn: the region after initial Hooke's region and yield point marked by arrows.

TABLE IV
Thermal Properties of Circular and Cruciform PET Filament

Cross-sectional shape	Mode	T_g (°C)	ΔH_{cc} (J/g)	$T_{cc,m}$ (°C)	ΔH_m (J/g)	T_m (°C)	α (%)
Circular	1st heating	[a]	−24.2	105.2	54.9	255.2	22
	2nd heating	80	–	–	51.5	248,5/255[b]	37
Cruciform	1st heating	[a]	−22.4	102.5	58.5	256.0	26
	2nd heating	80	–	–	51.8	246.7/253[b]	37

[a] Yarn overlapped.
[b] Shoulder.

shaped filaments shift to higher values ($m_1 = 27$, $m_2 = 114$). The fact that the plots show different slopes indicates that the failure is governed by different types of flaws. As expected, the circular shaped filaments show a continuous line for most of the filaments providing high strength and some artifacts in the lower strength range can be discarded. However, for the cruciform specimens, most of the single fibers tested fail at lower strength demonstrating the strong impact of surface defects. This indicates that the strength-controlling surface defects at the cruciform cross-sections of the fibers determine the filament strength resulting in lower average strength of 291 ± 19 MPa compared with 350 ± 18 MPa for circular shaped fibers. In other words, because of the rugged structure, the polymer heterogeneity is increased resulting in enhanced flaw severities.

The yarn tensile test results, according to the method discussed in the Tensile test section, are summarized in Table III. Each mean value reported is an average value of 10 separate measurements. Figure 6 illustrates average tenacity-elongation curves of 10 measurements of PET filament yarns manufactured from circular and cruciform cross-sectional shaped filaments spun at 3500 m/min. As clearly seen, the type of cross-section shape has a significant influence on tenacity and elongation at break. The strength of cruciform filament yarns is less compared to that of circular ones. Similarly, circular shaped filament yarns reveal higher elongation at break than cruciform filament yarns. The difference of elongation at break between circular and cruciform filament yarns is around 31%.

Although the yarn manufactured from cruciform filaments has lower elongation at break than that of circular cross-sectional shaped, the strength of cruciform filament yarn is considerably lower than that of circular filament yarn, which can be explained in the context of filament geometry. The lower degree of regularity and compactness in cross-sectional geometry of cruciform filaments than those of circular filaments results in the reduction of their load bearing capacity and strength. A careful examination of the curves in Figure 6 revealed that the length of region marked by arrows (the region after initial Hooke' region and yield point) varies with the change of cross-section. The length of this region in cruciform filaments are less than that in circular filaments, which is an indication of slightly higher orientation in cruciform filaments compared to circular filaments.

Glass transition temperature (T_g), melting point (T_m), enthalpy of cold crystallization (ΔH_{cc}) and melting (ΔH_m), and degree of crystallinity (α,%) obtained by DSC are detailed in Table IV showing that the type of cross-sectional-shape does not effect T_g and T_m of PET filament yarns. It can be seen that there is a difference in cold crystallization enthalpy during 1st heating of different PET filament yarns, which is the result of the different orientation degree during melt spinning. The greater the orientation degree, the lower is the cold crystallization enthalpy, as expected.

The degree of crystallinity of PET filament yarns determined by dividing the enthalpy difference (ΔH) from the 1st and 2nd heating step by the enthalpy of fully crystalline PET are termed as cold and maximum degree of crystallinity, respectively. Where,

$$\Delta H = |\Delta H_m| - |\Delta H_{cc}| \tag{4}$$

The enthalpy of fully crystalline enthalpy was taken as 140 J/g.[5]

As the crystallinity degree of as spun PET filament yarns of cruciform cross-section is slightly higher

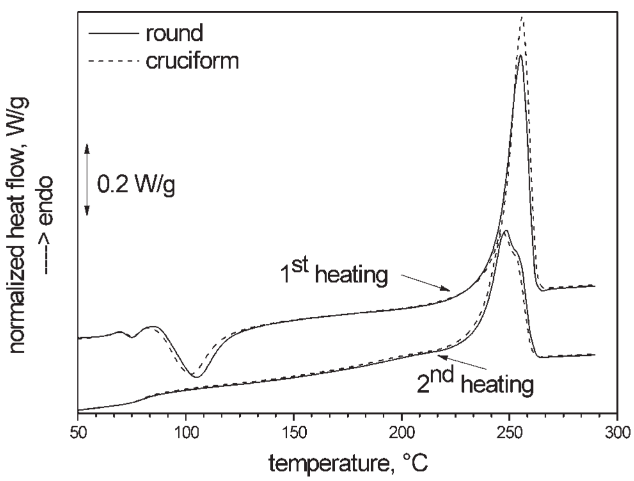

Figure 7 DSC curves of circular and cruciform PET filament yarns during 1st and 2nd heating.

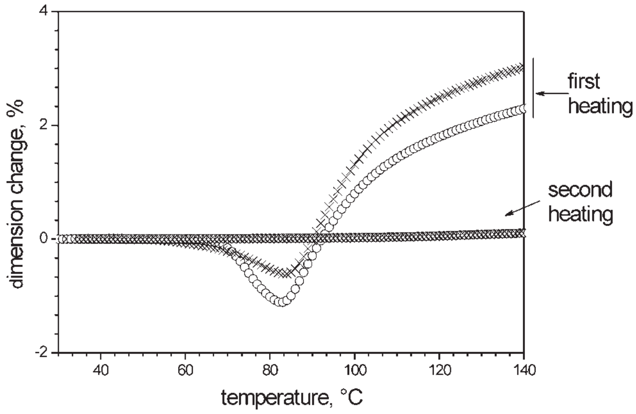

Figure 8 TMA curves of PET filament yarns with circular (○) and cruciform cross-sectional (**X**) shaped filaments during first and second heating under standard mode.

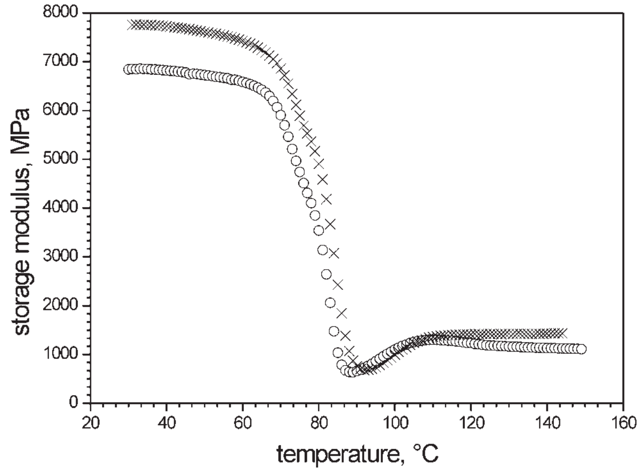

Figure 10 Storage modulus of PET circular (○) and cruciform (X) cross-sectional shaped filament yarns in dynamic mechanical thermal mode (DMA).

than that of circular cross-section ones and the maximum crystallinity of both cross-section is almost the same, it can be inferred that the shape of cross-sections does not significantly effect the crystallinity degree of undrawn PET filament yarn. Figure 7 shows the DSC curves of circular and cruciform PET filament yarns during 1st and 2nd heating.

Figure 8 displays the thermal behavior of circular and cross-sectional shaped PET filament yarns during first and second heating from TMA measurements under standard mode. Although the shrinkage starts earlier in cruciform filaments compared to circular filaments, their total shrinkage is less than that of the circular ones during first heating. However, during second heating, there is no difference in dimension change for both filaments due to the release of stresses after first heating.

Figure 9 shows modulated TMA curves of circular and cruciform cross-sectional shaped PET filament yarns. The total dimension change is further resolved into reversing dimension change and nonreversing dimension change, as described in Thermo-mechanical analysis section. It is clearly seen that dimension changes above glass transition temperature—from both reversing and nonreversing signals—of PET filament yarns with circular cross-sections are more pronounced under temperature influence than that of cruciform filaments. It can be attributed to less orientation of circular filaments compared to those with cruciform cross-sectional shape. From nonreversing signal, the shrinkage of cruciform filaments is higher than that of circular ones, indicating more internal stresses because of technological reasons. Agreeably, rather negligible dimensional changes (~ 1%) observed for both circular and cruciform filaments below the glass transition temperature reveal similarity in their physical characteristics.

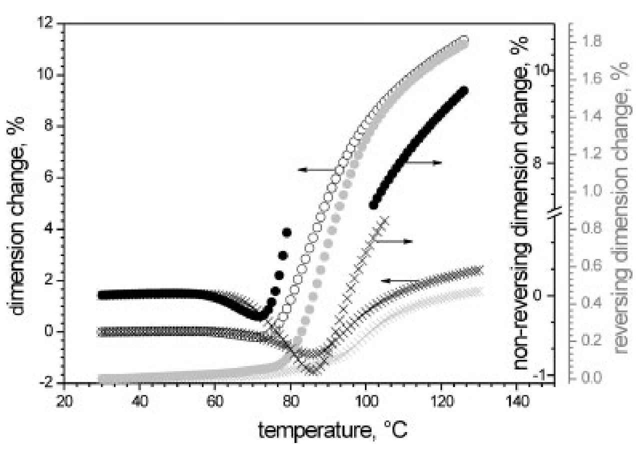

Figure 9 Modulated TMA curves of PET filament yarns of circular (○) and cruciform (X) cross-sectional shaped filaments.

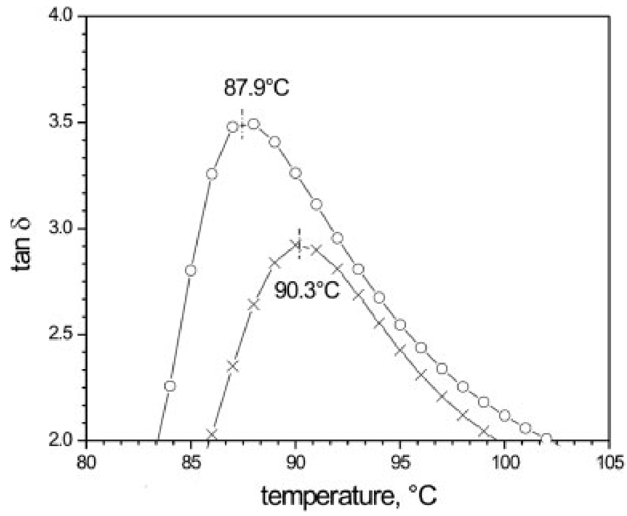

Figure 11 Tan δ of PET circular (○) and cruciform (X) cross-sectional shaped filament yarns under dynamic mechanical thermal mode (DMA).

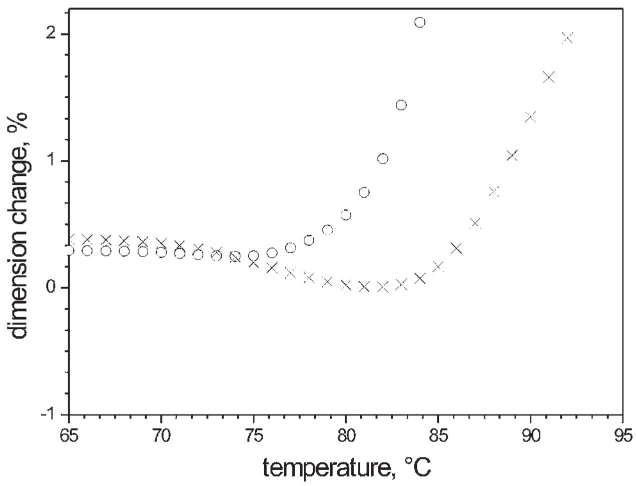

Figure 12 Dimension change of PET filament yarns of circular (○) and cruciform (X) cross-sectional shape of filament yarn under dynamic TMA mode.

Dimensional changes above the glass transition temperature of PET filament yarn spun are less important with respect to performance characteristics of fabrics manufactured out of them (washing, ironing etc.).

Figure 10 and Figure 11 illustrate differences in the temperature dependence of storage modulus and tan δ between PET circular and cruciform cross-section shaped filament yarns. T_g values for different filament yarns were estimated from the tan δ maximum. The tendency is observed that T_g is lower in the case of circular filaments in comparison to the cruciform ones. As tan δ value varies with frequency,[10] the value shown (cf. Fig. 11) is a relative value. The dimension change under dynamic TMA is shown in Figure 12, which is similar to the dimension change found using TMA under standard mode.

CONCLUSIONS

The influence of the filament cross-sectional type on the mechanical and thermomechanical properties of PET filament yarn is summarized on the basis of results discussed above.

The higher surface area per length (about 60%) of cruciform filament compared to that of circular filament leads to their higher cooling rate during melt spinning. Consequently, the cruciform filaments reach the final winding speed earlier associated with the shortening of the deformation zone leading to their increased orientation. The proof of their improved orientation can be found in their mechanical and thermomechanical properties:

- The cruciform filaments have lower elongation at break (by about 31%), although the strength of cruciform filaments is considerably lower than that of circular filaments arising from their lower degree of regularity and higher surface defects. In other words, because of the rugged structure, the polymer heterogeneity is increased in case of cruciform filaments resulting in enhanced flaw severities.
- Moreover, the cold crystallization enthalpy of cruciform filament yarns is lower than that of circular ones and crystallinity degree of cruciform filament yarns after manufacturing is slightly higher than that of circular filaments.
- The TMA results show that the cruciform filaments are less prone to dimensional change under the influence of temperature.

Although the results of DSC measurement do not show any difference in T_g for two different cross-sections of filament yarns, a tendency of higher T_g is found in the case of cruciform filament yarns from DMA measurements.

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