Effect of Process Variables on Bulk Development of Air-Textured Poly(trimethylene terephthalate) Bulk Continuous Filaments

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ABSTRACT: Bulk development of air-textured poly(trimethylene terephthalate) (PTT) bulk continuous filaments was studied by varying two texturing parameters, yarn preheating and texturing hot air temperatures. The yarns were subsequently heat treated from 80 to 160°C. Bulk was found to go through a maximum with increasing heat-treatment temperature because of two competing mechanisms. Upon heat treatment, the fiber shrunk and developed bulk; heat treatment also simultaneously induced structural reorganization through annealing and stabilized the fiber against shrinkage. When the later mechanism became dominant, bulk development decreased with further increase of heat-

treatment temperature. The temperature at which the maximum occurred increased when the yarn preheating or texturing air temperatures were increased. Depending on the extent of annealing and structural reorganization during yarn preheating and during texturing, fibers with equivalent bulk measured at a single temperature did not behave the same way over a range of heat-treatment temperatures. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 92: 1011–1017, 2004

Key words: polyesters; fibers; annealing; thermal properties; structure

INTRODUCTION

Poly(trimethylene terephthalate) (PTT) is a newly commercialized aromatic polyester. It is made by the melt polycondensation of 1,3-propanediol with either terephthalic acid or dimethyl terephthalate. One of PTT's major applications is in carpets^{1,2} because PTT carpets were found to have excellent elastic recovery, walk performance, low electrostatic propensity, and were resistant to acidic stains. Because of the polymer's low moisture absorption and fiber modulus, PTT carpets also have a desirable dry and soft feel.

Two kinds of synthetic fibers are used for making carpets: staple fiber and bulk continuous filaments (BCF). The use of BCF is growing, and the volume has now exceeded that of staple fibers because of the increasing number of carpet mills that are back-integrating carpet manufacturing into fiber extrusion. Virtually all of the newly installed machineries are intended for BCF because of the flexibility for making different types of yarns compared to staple fiber production. PTT BCF is made by extruding the polymer at a melt temperature of 250–265°C into multiple filaments that are cooled by cold air in a quench cabinet. The solidified multifilaments are then taken up by a haul-off roll, drawn between two pairs of godets, and texturized into BCF with pressurized hot air or steam, as shown in Figure 1.^{3,4}

Texturing is a process of giving bulk to the BCF. Bulk gives the fiber a lofty feel and provides coverage and body to the carpet. It also affects the carpet's walk performance and aesthetic; therefore it is an important property to control during BCF manufacturing. In general, the bulkier the BCF, the better is the carpet coverage because it takes less amount of fiber to give the same appearance of fullness than that of a lower bulk fiber. However, this comes at the expense of reduced carpet walk performance and a less-desirable aesthetic. Yarn tips of a cut-pile carpet made with high bulk BCF tend to look a little fuzzy.^{5,6} Thus, there is a balance between BCF bulk, carpet performance, and aesthetic in carpet manufacturing.

In a previous study,⁷ PTT BCF bulk development was found to depend on three texturing parameters: (1) the temperature of the godet used to preheat the drawn yarn [i.e., the Duo II godet temperature in the BCF machine (Fig. 1) used in this study]; (2) the texturing air pressure; and (3) the texturing air temperature. Changing these three parameters, either individually or their combinations, changes the yarn bulk. Although different combinations of these parameters can give the same measured bulk, attributed to the different heat histories in yarn preheating and texturing, BCF yarns of equivalent bulk do not necessarily respond the same way in downstream heat-treatment processes such as during heat setting and dyeing. If yarns of different heat histories were used in the same

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Figure 1 Schematic of a BCF machine and the yarn paths for drawing and texturing.

carpet they could cause uneven dyeing and bulk defects in the carpets. In this article, we studied the effect of texturing heat histories on PTT BCF bulking behavior as a function of heat-treatment temperature.

EXPERIMENTAL

BCF preparation

PTT [Corterra[®] polymer CP509200 (Shell Chemical Co., Houston, TX), intrinsic viscosity = 0.92 dL/g, measured in a 60/40 mixture of phenol and tetrachloroethane at 30°C] chips were dried in a circulating, dehumidified (-40° C dew point) hot-air drier at 130°C for 6 h to <30 ppm moisture. The dried chips were extruded at 252°C, drawn, and air-textured into 1500-denier 69-filament trilobal cross section BCF yarns using a Rieter Model JO-10 one-position, two-threadline pilot BCF machine, as shown in Figure 1. The setup of this machine is similar to the commercial production unit except it has only one position instead of a bank of four to eight positions. Therefore, the results of this study can be scaled for commercial

production. The modification ratio of the trilobal fiber cross section was 2.3. The modification ratio is a measure of the degree of the fiber cross section departing from being round. It is defined as the ratio of the diameter of a circle circumscribing the fiber cross section to the diameter of a circle inscribing the fiber cross section. The higher the modification, the more defined is the trilobal nature of the cross section. The extruded fibers were cooled and solidified in a quench cabinet (height: 1.7 m) with an 18°C cross-flow cold air at 0.4 m/s 1.0 wt % (by weight of the fiber) of a Limanol BF11L spin finish (Zimmer Schwartz) was applied to the filaments with a kiss roll. The fibers were taken up with an unheated haul-off roll, fed to a heated godet, called Mono I in Figure 1, and then drawn with a pair of heated godets, called Duo II, in an enclosed box with a see-through window. The drawn yarns were then textured with 7-bar pressurized hot air using a Rieter texturizing jet (model 616/1018-1662). The textured yarns formed a dense plug on a rotating perforated drum where they were cooled with inward flowing ambient air. The cooled yarns were then wound into approximately 6-kg packages with a traverseguide winder.

Table I shows the drawing and texturing conditions used in the study. Godet speeds and draw ratio were kept constant. The Mono I temperature and texturing air pressure were also kept constant at 52°C and 7 bar, respectively, whereas the Duo II temperature was varied from 120 to 180°C in 10°C increments, and the texturing temperature was varied from 140 to 200°C in 20°C increments.

Yarn bulk measurement

Bulk was measured using a modified ASTM D 4031-95a test method. Instead of using a fixed 120°C oven temperature, the temperature was varied from 80 to 160°C in 10°C increments to study the effect of heattreatment temperature on yarn bulk development.

TABLE I Drawing and Texturing Conditions for Making the PTT BCF	
Condition	Value
Speed (m/min)	
Haul-off Roll	884
Mono I	925
Duo II	2800
Draw Ratio	3.17
Temperature, °C	
Haul-off Roll	Unheated
Mono I	52
Duo II	120-180
Texturing air	
Pressure, bar	7
Temperature, °C	140–200

The term "bulk," used by the U.S. carpet industry, is called bulk shrinkage in the ASTM method. It is made up of two components: crimp imparted by the texturing process and the entropic shrinkage of the oriented fiber when it is heated to the test temperature. In this article we followed the industrial terminology of just calling it bulk.

A skein of 25,000-denier yarn was reeled from the yarn denier creel to the nearest number of revolutions. The initial length of the skein (L_0) was measured after applying a 50-g load, equivalent to an approximately 0.001 g/d tension. The yarn was placed in a circulating hot-air oven for 5 min. The skein was then removed and cooled for 1 min at room temperature. The same load was applied and the final length L_1 was measured after 30 s. Percentage bulk is calculated by

% Bulk =
$$\frac{L_0 - L_1}{L_0} \times 100\%$$

Measurements were done in triplicate.

RESULTS AND DISCUSSION

In a previous study, Werny and Chuah⁷ found that yarn preheating temperature (equivalent to the Duo II godet temperature in this study), texturing air temperature, and air pressure were the three main process variables affecting the BCF bulk. It is well known that when a fiber is subjected to a heating process, the heat history of the fiber plays an important role in its property and structure development. Because texturing is a thermomechanical deformation process, the texturing heat history is expected to be important to the BCF bulk development. Such an effect was not considered in the above study. In this article, we explore the effect of texturing temperatures on yarn bulk development when the fibers are heat-treated at different temperatures.

Before discussing the results, it is instructive to examine the thermal and mechanical steps involved in BCF texturing. The drawn yarn is first heated to above its glass-transition temperature (T_{q}) so that it becomes compliant and is easier to deform. It is then fed to the texturizer, which is a conical tube with an exit diameter slightly larger than the entrance diameter. A hot, high-pressure fluid such as air or steam is injected to the tube near the entrance. The fluid flow must be turbulent⁸ with a Reynolds number (Re) \geq 2000 to create vortices to separate the entering multifilament fiber bundle into individual filaments. As the filaments travel down the texturizer, they randomly impinge against the tube wall because of the turbulent airflow. They also impinge on the slower moving plug of textured fibers at the exit of the texturizer. These deformations cause the fibers to form random loops

and curvilinear configurations, called crimps. This section of the tube where such deformations take place is called the bulking chamber.

The effect of air pressure in texturing is rather straightforward. Zu and Wang⁹ analyzed the axial force (F_a) acting on an individual filament from the turbulent flow, and found it to depend on u^2 , where uis the air jet velocity. Given that *u* is a function of air pressure from Bernoulli's equation for airflow, therefore the higher the texturing air pressure, the higher the values of u and F_a to deform the fiber during texturing. For a BCF texturizer with a 3.5-mm tube diameter, the average *u* and Re were calculated to be 165 km/s and 2.1 \times 10⁵, respectively, when a 670-kPa air was used.¹⁰ It was also estimated that only about 7-10% of the air's kinetic energy is used to texture the fibers.⁸ The effect of air pressure in texturing is therefore mechanical in nature. BCF bulk increases with increasing air pressure because of a higher axial force for deformation and an increase in the frequency of the fiber impinging on the bulking chamber wall and the slower moving textured fiber plug.

The main purpose of Duo II preheating is to lower the modulus of the fiber such that it becomes easier to deform. Thus one would expect the fiber bulk to increase with increasing Duo II temperature. However, the effect is more complicated because other thermal events, such as annealing, orientation relaxation, and structure reorganization, occur simultaneously. The texturing hot air has dual functions. One is to continue heating the incoming fiber; the other is to set the textured fibers in their new configurations. Because air is a poor heat conductor, steam is sometimes used instead. Thus Duo II preheating, hot air temperature, and the residence time of the textured fiber in the bulking chamber determine its thermomechanical deformation history and bulk response to the subsequent heat-treatment process.

Any one of the above three texturing variables or their combinations can be changed to affect the BCF bulk; however, each of these variables gives the fiber a different thermomechanical memory. Fibers made with these different variables may have the same bulk when they are measured at the standard test temperature, however, their actual bulk developments could be different when the downstream processing temperature differs significantly from the test temperature because of their different latent thermomechanical memories. These differences can cause (1) uneven dye uptake, which results in carpet dye streaks¹¹; and (2) bulk streak, which is an optical effect attributed to the differences in light reflectance of fibers with different bulk.¹² Both types of streaks constitute major defects in carpet manufacturing. Because bulk uniformity is critical in carpet manufacturing, this study shows why yarns produced under different conditions have to be

Figure 2 Effect of heat-treatment temperature on PTT BCF bulk development. Yarns were preheated with Duo II godet temperature from 120 to 180°C and textured at a constant hot air temperature of 165°C.

kept as separate lots even if their measured bulk is equal.

In this study, we kept the air pressure constant because the effect is mechanical in nature, and explored the relationship of Duo II preheating and texturing air temperatures on PTT BCF bulk development at various heat-treatment temperatures.

Figure 2 shows the effect of oven heat-treatment temperature on PTT BCF bulk development from 80 to 160°C, which covers a range of temperatures the yarn might encounter in downstream processing, such as in heat setting and dyeing. In each series, the yarn was preheated with a Duo II temperature from 120 to 180°C, in 10°C increments, while the texturing air temperature was kept constant at 165°C. At each oven heat-treatment temperature, BCF bulk increased with increasing Duo II temperature. However, the degree of bulk development at each heat-treatment temperature was different, indicating that the process of bulk regeneration is dependent on the Duo II temperature. For each Duo II temperature, bulk went through a maximum with increasing oven heat-treatment temperature. To determine the temperature at which the maximum occurred (T_{max}) , the curves of Figure 2 were fitted by multiple linear regressions and T_{max} values were determined from the derivatives of the fitted equations. At 120°C Duo II, T_{max} was 66°C. As the Duo II temperature was increased, T_{max} also increased and was about 60°C lower than the preheating Duo II temperature. When the Duo II temperature was above

170°C and was higher than the 165°C texturing air temperature, T_{max} began to decrease (Fig. 3).

In a nylon 66 carpet staple yarn twist-setting study, Miller¹³ found the staple yarn bulk also went through a maximum with increasing heat-treatment temperature. The small-angle X-ray scattering (SAXS) intensities and the long periods were also found to increase monotonically with increasing heat-treatment temperature, indicating that the crystalline structures were annealed to more perfect and larger lamellar crystals. The occurrence of the maximum was explained as follows. After texturing, the yarn was wound into packages. The winding tension caused the yarn to lose bulk by pulling out some of the crimps. With stress relaxation over time, the yarn appeared to revert back to near its original configuration before texturing. The straightened yarns were said to be "cold set."14 The crimp pull out was temporary, however. The extent of cold setting depended on the time and temperature at which the textured yarn was held in that new straightened configuration. When the yarn was reheated to above its T_{α} during heat treatment, there was an onset of segmental molecular chain motion. The frozen-in cold-set strain was released; the fiber shrunk and regenerated the crimps or bulks depending on the extent of its latent thermomechanical texturing memory. The higher the reheating temperature, the more effective it was to regenerate the bulk. Thus fiber bulk increased with increasing heat-treatment temperature.

Besides the entropic driven shrinkage to regenerate bulk, there were simultaneous competitive enthalpic annealing and recrystallization processes occurring during heat treatments as observed by the increase in the SAXS intensities and the long periods. The increase in size and the perfection of the lamellar crys-



Figure 3 Effect of Duo II preheating on PTT BCF maximum bulk development temperature T_{max} .





Figure 4 Effect of Duo II preheating on PTT BCF bulk development at various heat-treatment temperatures.

tals helped set and stabilize the fiber against further shrinkage. Above a certain heat-treatment temperature, the kinetics of crystal perfection for stabilization became faster than fiber shrinkage in regenerating bulk; the fiber then was not able to fully recover the bulk. Therefore bulk decreased with a further increased in the oven heat-treatment temperature, and explained the maximum in the curves of Figure 2.

The increase in T_{max} with Duo II temperature of Figure 3, however, was attributed to in situ annealing and structural reorganization at the Duo II godets. Although the primary function of Duo II preheating is to lower the modulus of the fiber so that it becomes easier to texture, the heating process is always accompanied by some degree of annealing and structural reorganization depending on the temperature and residence time of the fiber at Duo II. As Duo II temperature increased, the degree of *in situ* annealing and stabilization of fiber against shrinkage also increased. It would therefore take a higher oven temperature for the fiber to shrink and recover the bulk. When the Duo II temperature became too high, such as at 180°C, the oven heat-treatment temperature was insufficient to fully recover the bulk, and T_{max} decreased as a result.

Figure 4 shows the relationship between PTT BCF bulk and Duo II temperature at constant oven heat-treatment temperatures. For clarity, data are shown in 20°C intervals. At each heat-treatment temperature, bulk increased with Duo II temperature, but not linearly. In general, the plots are convex in shape at low

heat-treatment temperatures. The explanation of such observation is similar to that of Figure 3. At higher Duo II temperatures, the fibers were significantly annealed and were structurally more stable. The lower oven temperatures were not as effective in regenerating bulk; the plots therefore became convex with increasing Duo II temperature. When the oven temperature was sufficiently high, such as at 160°C, the curve became concave because this high heat-treatment temperature was effective enough to cause significant fiber shrinkage and regenerate bulk. In general, the increase in PTT BCF bulk with Duo II temperature is nonlinear and depends on the oven heat-treatment temperature.

Figures 5 and 6 show PTT BCF bulk development as a function of oven heat-treatment temperature when the texturing temperature was increased from 140 to 200°C, whereas the Duo II temperatures were kept constant at 140 and 160°C, respectively. The trends in the bulk development were similar to those of Figure 2. At each texturing temperature, bulk went through a maximum with increasing oven temperature. At each Duo II temperature, T_{max} also increased with increasing air texturing temperature (Fig. 7), except they did not show the maximum of Figure 3, given that the texturing air temperatures were higher than the highest oven heat-treatment temperature used in the study.

The similarity between Figures 2, 5, and 6 is to be expected. Although the primary function of the Duo II

Figure 5 Effect of oven heat-treatment temperature on PTT BCF bulk development. Yarns were hot air textured from 140 to 200°C and the Duo II preheating temperature was kept constant at 140°C.

is to preheat the yarn to facilitate deformation, as the yarn enters the texturizer, heating continues by the texturing hot air. Therefore, the air temperature and yarn duration in the texturizer also determine the final effective texturing temperature before the fibers are discharged to the cooling drum. At the same time, the process of annealing and structural reorganization begins with Duo II preheating. The degree of annealing at Duo II then affects the ability of the hot air to texture



Figure 6 Effect of oven heat-treatment temperature on PTT BCF bulk development. Yarns were hot air textured from 140 to 180°C and the Duo II preheating temperature was kept constant at 160°C.

Figure 7 Effect of Duo II preheating on PTT BCF maximum bulk development temperature T_{max} with constant Duo II preheating temperatures of 140 and 160°C.

and set the yarn into its new configuration, and thus bulk regeneration during oven heat treatment. It is therefore difficult to separate the individual effect of Duo II preheating and texturing air temperature on PTT BCF bulk development. Nevertheless, the response of the BCF bulk to oven heat-treatment temperature showed that bulk depends on the relative temperatures of Duo II and texturing air. The texturing heat history then becomes important for yarn bulk development in the subsequent processing steps. Thus bulk measurement at a single temperature, such as the recommended 120°C for a polyester in the ASTM method, is inadequate to fully characterize the yarn, and as a quality control tool to prevent streaks from occurring in the final carpet.

CONCLUSIONS

PTT BCF bulk development during heat treatment is a function of the Duo II yarn preheating temperature and the texturing air temperature. Bulk increases and goes through a maximum when the heat-treatment temperature is increased. The temperature at which the maximum occurs increases with increasing Duo II temperature and decreases when the Duo II becomes higher than the texturing air temperatures. The heattreatment temperature required to develop the bulk also depends on the extent of fiber annealing and structural reorganization during Duo II preheating and texturing. Because the profile of bulk development depends on the thermomechanical history of





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- Duo II preheating and texturing air temperature, fibers with equivalent bulk measured at one temperature do not behave the same way over a range of heat-treatment temperatures experienced in the subsequent processing steps. Such measurement is therefore inadequate to fully characterize the yarn and to ensure bulk and dye uniformity in the carpet if the yarns were produced under different texturing conditions.

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