(REFEREED RESEARCH)

TWIST SETTING TEMPERATURE AND TIME EFFECTS ON MORPHOLOGY OF POLYETHYLENE TEREPHTHALATE YARN

BÜKÜM FİKSESİ İŞLEMİNDE SICAKLIK VE SÜRE PARAMETRELERİNDEKİ DEĞİŞİMİN POLİETİLEN TERAFTALAT İPLİĞİNİN ÖZELLİKLERİNE ETKİSİ

Kenan YILDIRIM[1], **A. Melek KÖSTEM**[2], **Nurcan AYDIN**[2], **Hüseyin TUNA**[2], **Ayhan CENAN**[2]

[1]Bursa Technical University, Department of Fiber and Polymer Engineering, Bursa, Turkiye
[2]TÜBİTAK-BUTAL Bursa Test and Analysis Laboratory, Bursa, Turkiye

Received: 01.11.2013 Accepted: 04.06.2014

ABSTRACT

The twist on the yarn should be fixed by heat to remove twist liveliness. This procedure changes the properties of polyethylene terephthalate (PET) yarn in aspect to both fiber morphology and yarn mechanical behavior. The yarn properties are affected by heat setting process factors which mainly are heat-setting method, temperature and time. This study investigates the effect of temperature and time of the heat setting processes on the yarn properties. The change of the morphology of the yarn was determined by tensile test, differential scanning calorimetric (DSC) analysis, boiling water shrinkage test, bobbin hardness and twist amount per unit length test. The study demonstrates that the time of the heat setting does not have a considerable effect on the PET yarn properties. The temperature of the heat setting has dominant influence onto the properties of the PET yarn, especially on the boiling water shrinkage and tensile behavior. The tensile strain and bobbin hardness increase, and shrinkage decrease with increasing heat setting temperature. The force-strain curve shape is changed by altering twist setting parameters. These results show us that heat setting temperature related to stress relaxation arise from twist insertion, rearrangement of the macromolecules and molecular orientation in the amorphous region.

Key Words: Heat setting, Twist fixation, PET filament, Structure–property relationships.

ÖZET

İpliğe verilen bükümün sabitlenmesi için ısıl işlem gereklidir. Bu işlem polietilen teraftalat (PET) ipliğin hem morfolojisinin hem de mekaniksel özelliklerini değiştirmektedir. İplik özellikleri ısıl işlem faktörlerinden ısıl işlem metodu, sıcaklık ve süreden büyük ölçüde etkilenmektedir. Bu çalışmada ısıl işlem sıcaklık ve süre parametrelerinin iplik özellikleri üzerine etkisi incelenmiştir. İplik özelliklerinin değişimi çekme testi, diferansiyel taramalı kalorimetre (DSC), kaynama çekmesi, bobin sertliği ve büküm miktarı testleri ile belirlenmiştir. Isıl işlem süresindeki değişimin özellikle kaynama çekmesi ve çekme özellikleri olmak üzere iplik özellikleri üzerine büyük oranda etkisinin olmamasına karşın, sıcaklığın etkisinin yüksek olduğu görülmüştür. Isıl işlem sıcaklığının düşmesiyle kopma uzama oranı ve bobin sertliğinin arttığı, kaynama çekmesinin azaldığı görülmüştür. Bu sonuç ısıl işlem sıcaklığının ipliğe verilen bükümden dolayı oluşan iç gerilimlerin giderilmesi, iç yapıdaki moleküllerin yeniden yerleşimi ve amorf bölgedeki moleküler oryantasyon ile yakından ilgili olduğunu göstermiştir.

Anahtar Kelimeler: Isıl işlem, Büküm fiksesi, PET filament, Yapı-özellik ilişkisi.

Corresponding Author: A. Melek Köstem, melek.kostem@tubitak.gov.tr, Tel: +90 0224 233 94 40

1. INTRODUCTION

Twist is giving a form to a yarn by relative rotation of the yarn ends to bind the fibers or filaments together. This process like ring twisting, open-end spinning, self-twist spinning, false-twisting, two-for-one twisting or uptwisting generates tension in the yarn. During the unwinding of the bobbins, to facilitate decrease in the tension on the yarn, a reverse direction twist occurs, resulting in forming of snarling. Spirality of knitted fabrics and skewness of woven fabrics are also attributed to the level of snarling, result from twist liveliness of the yarn. Before using the yarn, the twist on the yarn should be fixed via conditioning or heat setting to overcome these problems via eliminating of stress in the yarn (1-3).

Industry uses dry air, contact heating system, water vapor, or liquid baths as heat setting processes. The heat setting changes the structure, morphology and physical properties of the PET yarn. Stress-strain, recovery behavior, dye-uptake, optical properties and thermal properties of PET yarn are affected by heat setting parameters(4). Any increase in the heat setting temperature decrease the thermal shrinkage of PET yarn via releasing more stress from its molecular structure. According to the study carried out by Barham and Keller, the fibrous structures have different behavior before and after drawing and rolling process with respect to thermal shrinkage. Unlike the fibrous structures which are not drawn and rolled, the fibrous structures which are drawn and rolled in solid state shows thermal shrinkage during heated close to melting point without converting them into molten state. For the reason that, crystals are formed due to stress and flow induced crystallization during the polymerization, and these crystals are disrupted and molecular chains are aligned in the direction of fiber during solid state drawn procedure result in locked-in stress on the tie molecules spanning between crystallites (5). According to their study on the PET fiber before and after drawn and heat setting Gupta et al. claimed that the locked-in stresses were responsible for dimensional instability. The fixing of twist on the yarn for getting of rid of snarling via heat setting can be explained on the same basis as the relaxation of residual stresses and thereby setting the shape (6). According to the study carried out on the PET film, there are close relationships between heat-setting, crystallinity ratio, and drawing process. The drawing procedure generates stress-induced crystallization in PET films. As a consequence, the crystallinity ratio in the drawn PET film is much higher than the crystallinity ratio in the undrawn PET film in both cases of heat settings which are done below and upper 100^{o}C. The crystallinity ratio increases in PET film and yarn with increasing heat setting temperature in both cases of drawn and undrawn (7, 13). During heat setting, it is considered that the crystalline morphology is changed through a number of routes such as partial melting and recrystallization, crystallization of oriented amorphous phase, perfection of crystals, thickening of crystals, entanglement slippage, and chemical healing (4). According to a study carried out on the twisted Polyamide (PA) filament yarn in case of heat setting by dry air, the fractional recovery of twist decrease and crystallinity ratio, crystallite size and crystalline perfection increase with increasing heat setting temperature. Unlike the increasing α and γ crystal height, tensile strain of the yarn decreased with an increasing heat-setting temperature in constant time. Dye absorption behavior and strength-strain graphic shape of the yarn also changed by heat setting temperature especially above 100^{o}C (4,8,9). PET yarn is also sensitive to heat setting parameters with respect to dye uptake. The dye uptake decreases with increasing heat setting temperature. The decreasing level depends on the dyestuff type and dying methods (13).

Both knitted and woven fabric manufacturers sometimes encounter a problem as a consequence of inaccurate heat setting parameters. This type of fault on the woven fabric is given in Figure 10. This type of fault occurs when using twisted filament yarn from FDY (fully drawn yarn) yarn by using two for one twist method. This fault looks like the fault which arises from using PET yarn with different or faulty production parameters.

This study involves the effect of both heat setting temperature and time on the PET filament yarn for the case of dry air heat setting. The effect of the change of the heat setting parameters on the yarn properties was clearly proved by using data of tenacity, tensile strain, strength-strain curve, DSC thermogram, crystallinite ratio, boiling water shrinkage, bobbin hardness and twist amount per unit length.

2. METHODS AND MATERIALS

150 denier 96 filament fully drawn (FDY) polyethylene terephthalate (PET) yarns were used. The yarns were twisted by using two for one twisting technique. All twist parameters were kept constant. The twisted amount on the yarn was 550 (Z) T/m. The samples were subjected similar heat setting conditions by various temperature and time. The applied heat setting temperatures were 85^{o}C, 95^{o}C, 105 ^{o}C, 115^{o}C, 130^{o}C, and 145^{o}C and within time intervals of 30 min, and 60 min. After the heat setting all yarns were conditioned at room temperature for 2 months.

One-way ANOVA model with Student-Newman-Keuls (SNK) test was used in the experiments and results were analyzed accordingly by using SPSS version 12 statistical program. Test specimens were various according to parameter tested. Test numbers were given in relevant subtitle. All test results were analyzed at 0.05 significance level.

3. MEASUREMENTS

Thermal Behavior and Crystallinity Ratio

DSC is the most widely used equipment among all thermal techniques. During the heating of the sample in the DSC, molecular mobility in amorphous region, cold crystallization, partial melting, annealing, recrystallization and complete melting occurs. The crystallinity ratio in the semi-crystalline polymer changes the cold crystallization behavior with respect to change in enthalpy (heat of fusion) and peak temperature. The thermogram obtained from the first heating (as-received) stage involve information both about material features and thermal history such as production factors effect, but second heating (heating-cooling and heating) thermogram gives data only about material characteristic (14-16). Unlike indium which contains single crystal, polymers involve a range of crystals of varying stability that melt over a broad range. Due to that, calculation of onset temperature is fairly difficult. The maximum peak is the most meaningful information in practice.

Crystallinity ratio was measured according to DSC technique. The calculation of peak area on melting region according to Equation 1 is very helpful in process development and material characterization. The peak area can also be used to identify crystallinity ratio of material (10). The crystallinity ratio measurements were performed using a Perkin–Elmer Sapphire differential scanning calorimeter. 140.1mJ/mg was used for ΔH (fusion) for 100% crystalline PET. Two specimens have been tested for crystallinity ratio measurements for each temperature.

$$\text{Crystallinity ratio} = (\Delta H_m - \Delta H_c) / \Delta H_m^{\circ} \times 100\% \quad (1)$$

ΔH_m: The heat of fusion of melting (J/g)

ΔH_c: Cold crystallization (J/g)

ΔH_m°: The heat of fusion of melting if the polymer were 100% crystalline (J/g) [11,12,16].

For DSC Analysis Perkin Elmer Sapphire II model Differential scanning calorimeter (DSC) having heat flux method was used. Initial temperature was 30°C, final temperature was 350 °C, heating rate was 10°C /min, purge gaseous was nitrogen, sample mass was approximately 10 mg, sample pan was crimped aluminum pan, heating type was first heating (as-received).

Tensile Properties of the Multifilament Yarn

Tenacity and tensile strain of the yarn were tested according to ISO 2062 using CRE class 1 SDL universal tensile tester having 500 N load cells. Fifteen specimens were tested for each sample. The gauge length was 500 mm. The rate of extension was 500 mm/min. The maximum load was used for tenacity calculation. Tensile strain was measured at maximum load (17).

Twist amount per unit length

Twist amount per unit length test was performed according to ISO 2061 standard using open-closed method by zweigle D314 model tester. The sample length was 20 cm and the applied load was 8 g. Twenty specimens were tested for each sample. (18).

Boiling water shrinkage

Boiling water shrinkage test was carried out according to DIN 53866. Six specimens have been used. Tensioning weight of 0.125 cN/tex was applied to the yarn which was 1 m length and hank is formed. The first length recorded in this condition as l_1, and then the load was removed. The yarn was wetted in a soap solution (1 g soap/1 l water) and left in the solution at 100°C for 15 min, dried an hour at 60°C, hanged the device again and waited one hour. Then the same weight applied to the yarn and the length was recorded as l_2. Boiling water shrinkage was calculated from Equation (2).

$$\text{Boiling water shrinkage (\%)} = ((l_1-l_2)/l_1)\times 100 \qquad (2)$$

Bobbin hardness

Bobbin hardness test was done by using hand type Schmidt HP5 bobbin hardness tester by the way of directly measured on the bobbin. Five specimens were tested for each sample.

4. RESULTS AND DISCUSSION

The actual results are shown in Figures 1,4,5,7,8,9. The heat setting temperature dramatically affects the shrinkage properties and tensile strain of PET yarn. These two properties are related to the oriented and non-oriented amorphous phases of the yarn (19-22).

Any change on the temperature or time of the heat setting processes affects the crystallinity ratio of the PET yarn (Figure 1). There was a small difference in the crystallinity ratio depending on the heat setting condition except the condition at 105 °C for 30 min. According to the literature crystallization occurs when PET yarn is annealed above its glass transition temperature (Tg) without tension (21, 24), due to chain folding of macromolecules in the amorphous region. Tg temperature is related to bond breakage between macromolecules in the amorphous region (10, 23). According to study carried out by Statton et al. the amount of chain folding depends on the temperature and tension during heat treatment. Chain folding increases with increasing temperature and decreasing tension (25). The tension resulting from the twist on the yarn hinders crystallization in the heat setting procedure of PET yarn. When heat setting was done about or below Tg temperature, crystallization cannot be formed because of the fact that there is no chain bond breakage in the amorphous region. It is assumed that the crystallinity ratio decreased when heat setting was carried out at 105 °C for 30 min, since the macromolecule rearrangement starts but could not be completed due to short heating time. Therefore, some crystalline structure may be deformed. The deformation on the crystalline structure can be seen from DSC thermogram of the yarn which was treated at 105 °C for 30 min (Figure 3). The melting peak of this thermogram could not be formed properly. The right leg of the melting peak was shorter than the other one. This defect may arise from deformation of the crystalline structure, as there was energy drop on the thermogram parts related to melting of the crystalline regions. Similar situation can be seen on the DSC thermogram of the yarn which was treated at 130 °C for 30 min. However, the defect level was less than the defect level on the DSC thermogram of the yarn treated at 105 °C for 30 min. The smaller defect might result from the higher temperature which could break more bonds between macromolecules in the amorphous region to overcome high deformation on the crystalline structure. Contrary to the melting peak on the DSC thermogram of yarns treated for 30 min., the melting peak which belongs to the yarns treated for 60 min. was formed properly, and there was not any energy drop on the melting peak region for all the temperature (Figure 2, 3).

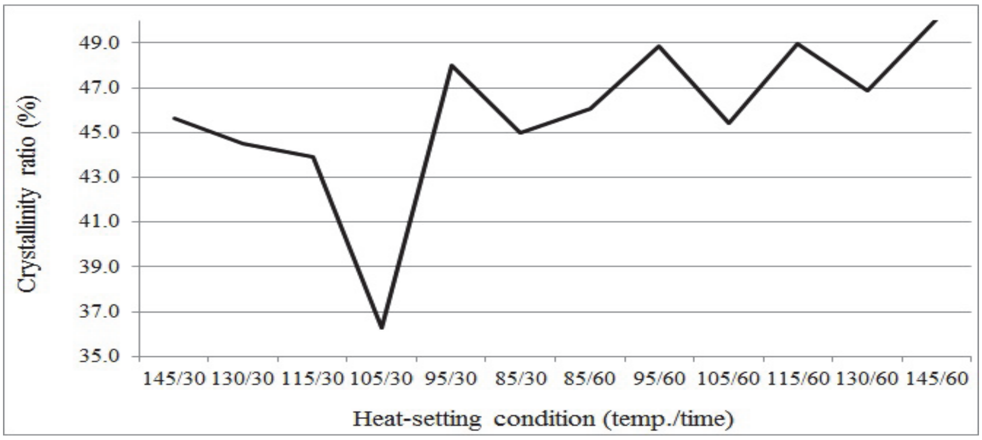

Figure 1. Crystallinity ratio of twisted PET yarns

Unlike the melting peak shapes, which is related to breakage of bonds between chains in crystalline region, the melting peak shapes of yarn treated for 30 min were not smooth compared to the yarns treated for 60 min. (Figure 2,3). Also some other defects between melting peak and Tg were observed in the thermogram of the yarns treated for 30 min. as opposed to the yarns treated for 60 min. These two facts also support our claim that a heating-time of 30 min. was not enough for the rearrangement of macromolecules in the amorphous region and proper crystalline structure formation.

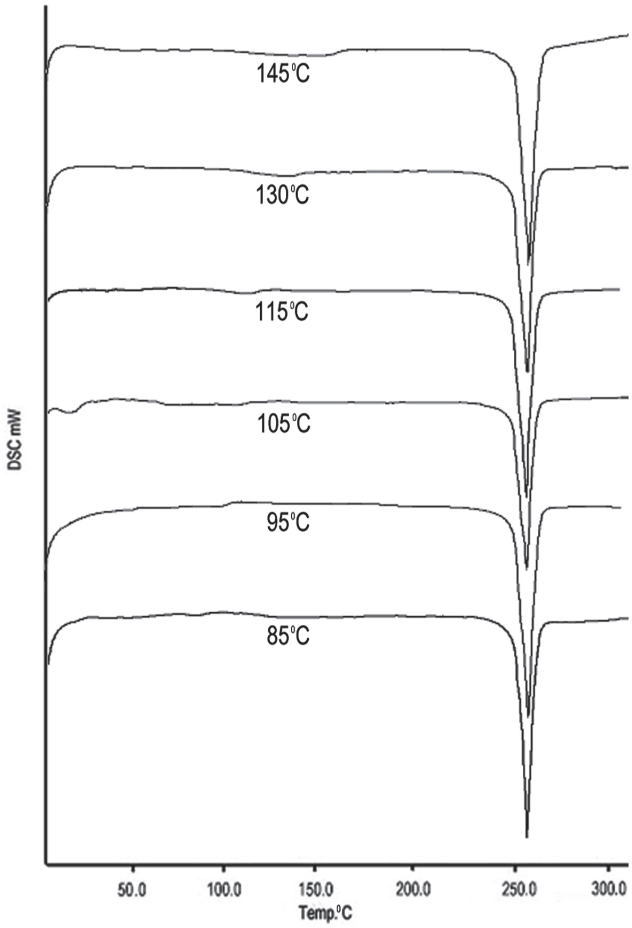

Figure 2. DSC thermograms of twisted PET yarns heat-setting was done in 60 min. at different heat-setting temperatures.

According to variance analysis and Student-Newman-Keuls (SNK) results evaluated at the 5% significant level, the hypothesis for time factor was accepted for all variables. The hypothesis for temperature factor was rejected for all variables except twist amount per unit length variable. The hypothesis for interaction of the factors was accepted for twist amount per unit length and tenacity variables. From that analysis, it can be concluded that there is a relationship between heat setting condition and PET yarn properties, heat setting temperature in particular. While the duration of heat setting has no affect on the PET yarn properties, the temperature has tremendously affect the PET yarn properties except twist amount. The duration of heat setting influences the morphology of PET yarn by causing improper macromolecule rearrangement and some crystalline structure deformation as seen in Figures 2,3.

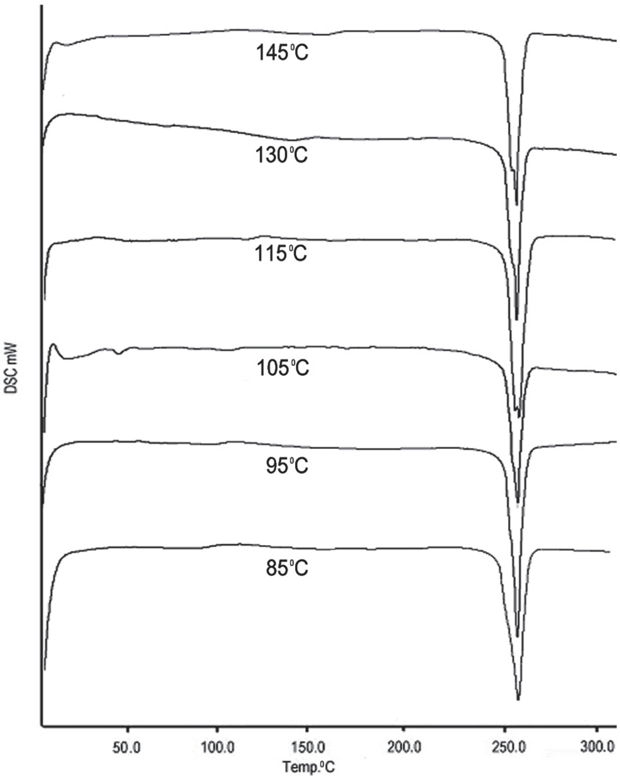

Figure 3. DSC thermograms of twisted PET yarns heat-setting was done in 30 min. at different heat-setting temperatures.

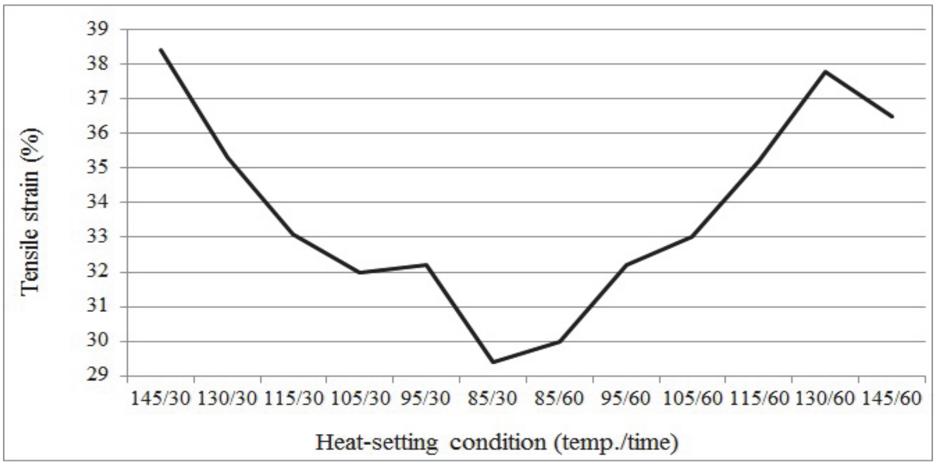

Figure 4. Tensile strain of twisted PET yarns

Tensile strain is affected **by** heat setting temperature. Unlike shrinkage, tensile strain and strength increase with increasing temperature (Figure 4,5 and 9). Similar to dye uptake, both of these properties are related to oriented and non-oriented amorphous regions. Shrinkage and tensile strain increase whereas tenacity decreases with decreasing orientation in the amorphous region if crystallinity ratio remains constant (26, 27). According to the tenacity and shrinkage test results, orientation in the amorphous region increases with increasing heat setting temperature. The conflict between literature and our results in case of tensile strain **arises** from twist generating stress in the yarn morphology. The twist generates stress in the yarn via torsion and bending forces. These forces are absorbed by chains in the non-oriented amorphous region, because these macromolecules have more mobility than the other molecules in the other regions. This stress inhibits mobility of macromolecules resulting in less strain. Decreasing molecular mobility decreases tensile strain. Removal of stress with temperature increment removes the hindrance on the chains mobility and increases the orientation in the amorphous region. The effect of stress elimination can be seen in Figure 6 on the stress-strain curve. An increase on the heat setting temperature increases the elasticity of the yarn in the region between 10-90 mm elongation region. This part is related to non-oriented amorphous region. Orientation in the non-oriented amorphous phase takes place in this region when PET yarn is under tensile test. When yarn is twisted, the macromolecules in the non-oriented amorphous region are affected mostly, oriented amorphous region and crystalline structure are not affected seriously due to rigidity of these structures. The mobility of chains in the non-oriented amorphous region is higher than the macromolecules in the oriented amorphous region and crystalline structure. Tensile strain depends mostly on non-oriented amorphous region. Any decrease in this region and any hindrance onto mobility of the macromolecules in this region decreases tensile strain. Therefore, the tensile behavior of twisted yarn in the region of 10-90 mm elongation looks like more rigid materials (Figure 6, curve: 85,95,105). During the heat setting above Tg temperature with adequate time, the bonds between macromolecules in the amorphous region are broken which results chains to obtain mobility for rearranging themselves to eliminate stress. After the removal of stress, PET yarn obtains again its elastic behavior (Figure 6, curve: 115/60, 130, 145). The results of boiling water shrinkage and tenacity show that the orientation in the amorphous region increases with increasing heat setting temperature. The increase on the tensile strain with increasing heat setting temperature indicates that the hindrance based on the orientation on the chain mobility effect is lower than the hindrance based on the stress.

Bobbin hardness increases with increasing heat setting temperature (Figure 7). The increase of bobbin hardness is also related to removal of stress and rearrangement of macromolecules as a result of increasing orientation. The bobbin hardness is also attributed to chain folding in the amorphous regions. Chain folding and rearrangement of macromolecules enforce the yarn to shrink. This shrinkage increases with increasing heat setting temperature. The increase in shrinkage leads to an increase in bobbin.

Although twist liveliness **is** affected seriously from heat setting condition (1-3), twist amount on the yarn is not affected from heat setting condition. Altering both heat setting temperature and time has not resulted considerable influence on the twist amount of yarn. As inconvenient heat setting process effects fixation of the twist on the yarn and twist liveliness which are important factors to overcome snarling, twist amount is not affected from heat setting condition (Figure 8).

Shrinkage decreases with increasing heat setting temperature (Figure 9). The decrease on the shrinkage is also related to orientation increase and elimination of stress resulting from rearrangement of chains. The result of the study carried out by Karakas shows that the increase of first heater temperature in the draw-texturing procedure decreased thermal shrinkage of PET yarn as a result of reduced residual stress and molecular mobility. Also the author claimed that the mobility of the polymer chains increased with increasing temperature resulting in ascending flexibility of the macromolecules and the motion of structural elements to easier alignment (29). Statton et al claimed that the increased number of chain folds during heat treatment lowers the thermal shrinkage that occurs subsequently in boiling water (25). PET yarns elongate instead of shrinkage at and above 130 ^{o}C heat setting temperature especially if heat setting time is 30 min. There are two reasons of this extension. It may be related to twist opening. It is known that twist opening causes extension of yarn length and vice versa. This extension may be related to melting of small size and defected crystals which are formed during heat setting. The crystalline region could not be formed properly due to short time. Therfore, these types of crystals have defect and can be deformed at low temperature annealing like 100 ^{o}C.

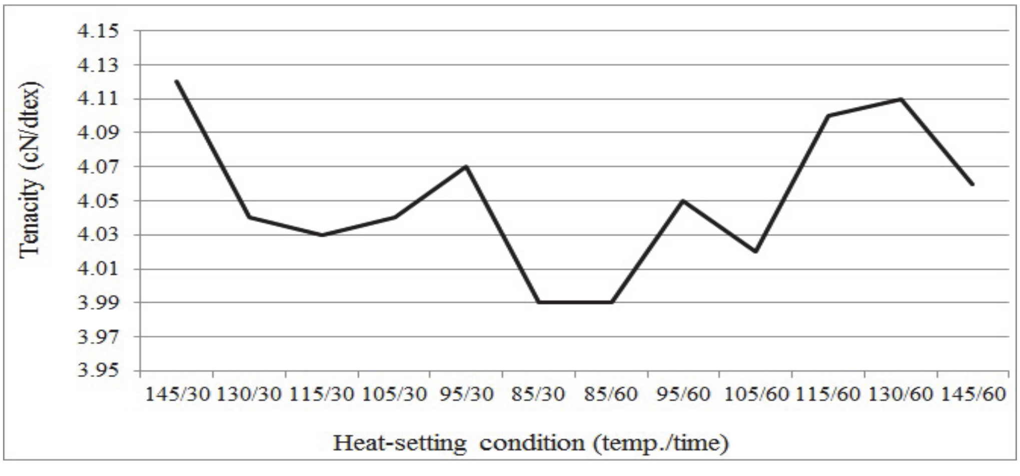

Figure 5. Tenacity of twisted PET yarns

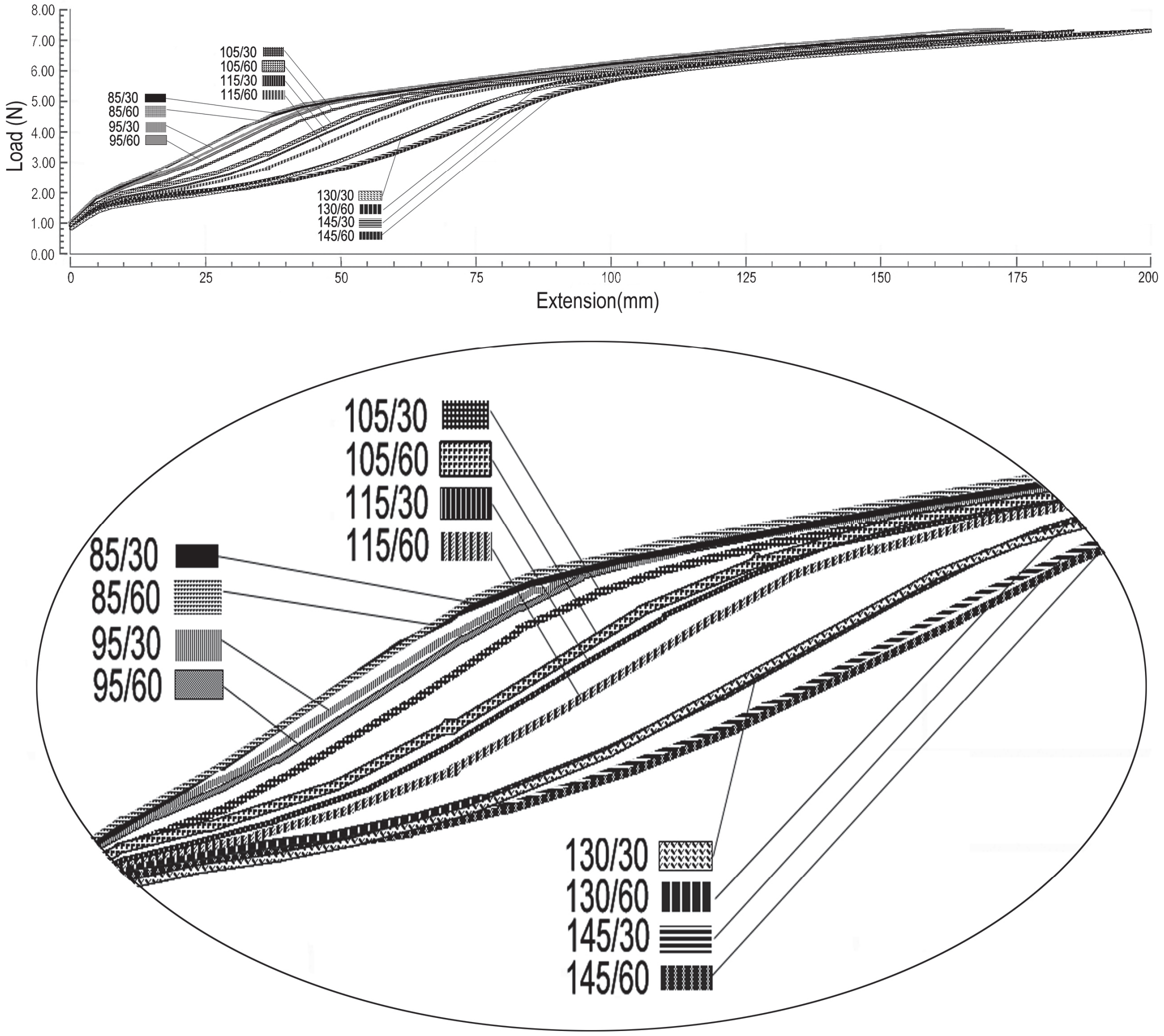

Figure 6. Load-extension curve of twisted PET yarns

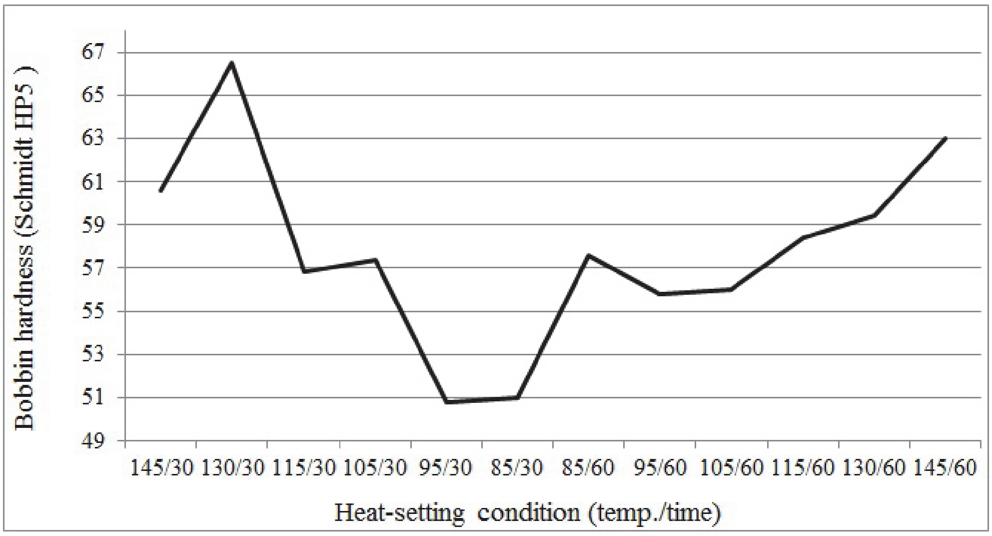

Figure 7. Bobbin hardness of twisted PET yarns.

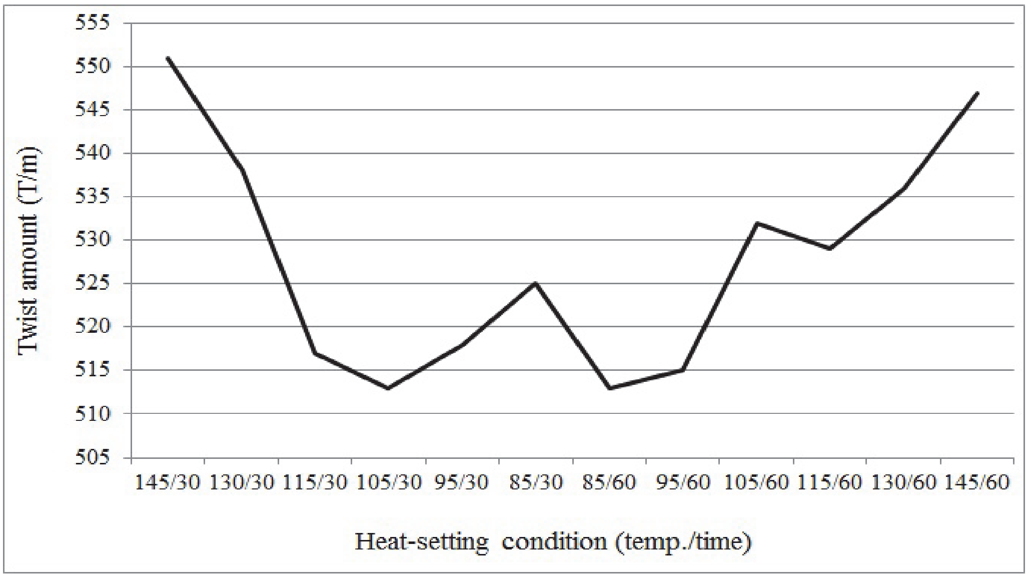

Figure 8. Twist amount of twisted PET yarns

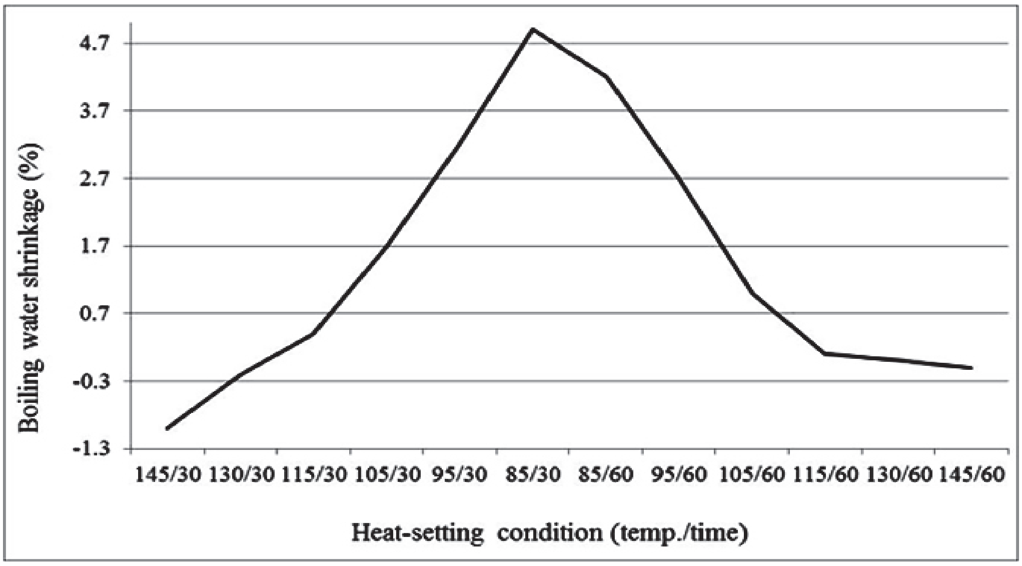

Figure 9. Boiling water shrinkage of twisted PET yarns

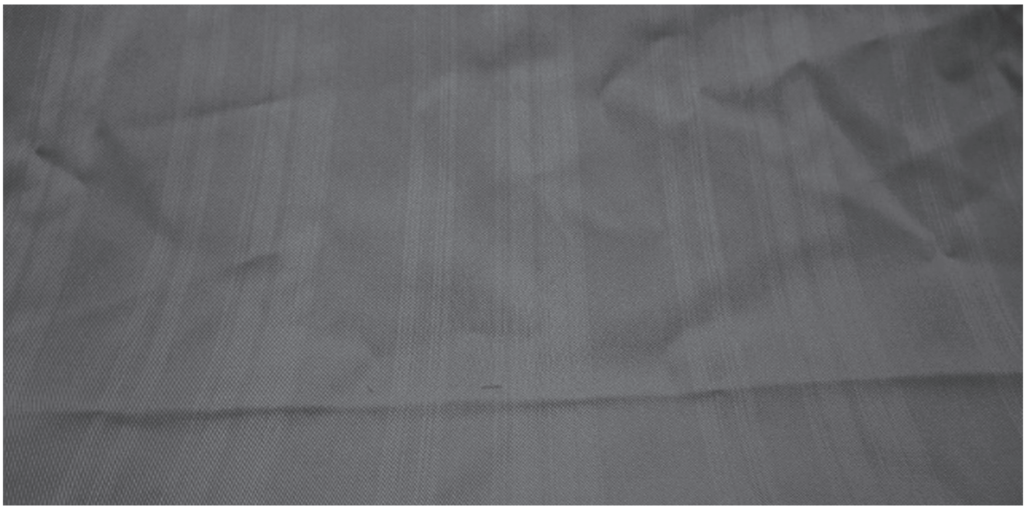

Figure 10. Fault on the fabric result from incorrect heat setting process parameters applied on the two for one twisted filament yarn (adapted from the reference of 30).

The fabric on Figure 10 was produced from same yarns which were identified in materials and method section with respect to FDY yarn properties and twist condition. Only heat setting condition was different. This fabric was produced commercially by Akınbay Tekstil firm. Warp preparation was done using these yarns that were heat set at two different heat setting temperature. The dark part of the fabric was formed with the yarns that were heat set at higher temperature than the yarns in the light part of the fabric. Fabric was colored by commercially HT dying procedure with disperse dyestuff (30).

5. CONCLUSIONS

According to data from DSC thermogram, load-extension curve and dying fault on the fabric, it can be concluded that the heat setting conditions dramatically influence amorphous regions. Tensile strain, boiling water shrinkage and tensile behavior of the yarn are seriously affected from the heat setting temperature. The increase of the bobbin hardness also supports that increasing heat setting temperature facilitates the mobility of the macromolecules. With increasing heat setting temperature, tensile strain and bobbin hardness increase, thermal shrinkage decreases and the elasticity of yarn increases. There was a morphological change of PET yarn depending on heat setting duration. The heat setting time influences the crystalline structure and chain rearrangement of the yarn. The heat setting time should be 60 min. or higher to prevent deformation in the crystalline structure and hindrance of molecular rearrangement. Dye uptake amount is related to amorphous regions rather than crystalline phase. Any increase on the orientation in the amorphous regions decreases dye uptake and increases birefringence (26, 28, 29). The reason for the dying fault shown in Figure 10 is using yarns which have different dye uptakes and different light reflection properties. Both of these properties depend on the orientation ratio in the amorphous region. The effect of the heat setting time is negligible with respect to dye uptake difference. The heat setting time is crucial for the crystalline structure, size and deformation. The temperature is a very dominant factor on the formation of this type of fault in the heat setting process. Similar result was found by Sardag et al. about cellulosic yarns setting by vacuum steaming (31). The orientation ratio in the amorphous region influences dye uptake and birefringence properties of the PET yarn. In conclusion, the heat setting temperature influences the morphology of the PET yarn with respect to orientation ratio in the amorphous region which results in dye uptake differences shown in Figure 10.

ACKNOWLEDGEMENT

Thanks to Akinbay Tesktil San. ve Tic. A.Ş for preparing twisted PET bobbin from FDY yarn.

REFERENCES

1. B. Xu, and C. Murrells, 2008 ,“Automatic Measurement and Recognition of Yarn Snarls by Digital Image and Signal Processing Methods”, *Textile Res. J.* **78**, 439.
2. S. Sardag, M. Kanık, and O.Ozdemir, 2012, “The Effect of Vacuum Steaming Processes on Physical and Dyeability Properties of Polyamide 6 Yarns”, *Textile Res. J.* **80**, 1531
3. S. Sardag and O. Ozdemir, 2012,“The effects of tandem and conventional vacuum steaming methods on the properties of yarns”, *Textile Res. J.* **82**, 181.
4. V. B. Gubta*,* 2002, “Heat Setting”, *J Appl Polym Sci*, **83**, 586 .
5. P. Barham, and A. Keller, 1975, “A criterion for distinguishing between polymer fibers of fundamentally different origin”, *J Polymer Sci Polymer Lett Ed*, **13**, 197.
6. V. B. Gupta, J. Radhakrishnan, and S. K. Sett, 1995,” Effect of process parameters on residual stress distribution in poly(ethylene terephthalate) fibers and films as revealed by chemical etching”, *J Appl Polym Sci*, **55**, 247.
7. H. Masanori, H. P. Chung, and A. Masanori*,* 1984*,* “Effects of Heat Treatment and Mechanical Stresses on The Dielectric Strength of Uniaxially Drawn Pet”, *IEEE T DIELECT EL IN* , **19,** 273
8. N. Vasanthan, 2004,“Effect of Heat Setting Temperatures on Tensile Mechanical Properties of Polyamide Fibers”, *Textile Res. J.*, **74**, 545
9. N. Vasanthan, 2007, “Effect of the Microstructure on the Dye Diffusion and Mechanical Properties of Polyamide-6 Fibers” *J. Polym. Sci. Part B Polym. Phys.*, **45**, 349
10. A.W. Salamon, K.J. Fielder “Practical Use of Differential Calorimetry for Plastics” In *Handbook of Plastics Analysis*, (H. Lobo, J.V. Bonilla, Eds.), p p.79-109, Marcel Dekker, New York, 2003
11. W .J. Sichina, DSC as Problem Solving Tool: Measurement of Percent Crystallinity of Thermoplastics, Perkin Elmer Instruments PETech 4*0*, Perkin Elmer Instruments
12. R. G. Matthews, A. Ajji, M. M. Dumoulin, and R.E.Prud’homme, 2000, “The effects of stress relaxation on the structure and orientation of tensile drawn poly(ethylene terephthalate)”, *Polymer,* **41**,7139
13. K. V. Datye, and A. A. Vaidya, Chemical processing of synthetic fiber and blends, pp.53-59, Wiley-Interscience, New York, 1984
14. M. J. Richardson, 1984, “Thermal analysis of polymers using quantitative differential scanning calorimetry”, *Polymer Testing*, **4**, 101
15. M.J. Forest, “Application to Thermoplastics and Rubbers” in Principles and Applications of Thermal Analysis, (P. Gabbott Ed.), pp.190-249, Blackwell Publishing, Singapore, 2008
16. Y. Kong and J. N. Hay, 2002, “The Measurement of Crystallinity of Polymers by DSC”, *Polymer*, **43**, 3873
17. ISO 2062, Determination of single-end breaking force and elongation at break using constant rate of extension (CRE) tester
18. ISO 2061, Determination of Twist in Yarns - Direct Counting Method
19. A. L. Volynskii, L. M. Yarysheva, and N. F. Bakeev, 2011, “Visualization of Deformation Induced Structural Rearrangements in Amorphous Polymers”, *Polymer Science, Ser. A,* **53**, 871
20. J. Shimizu, N. Okuı and T. Kikutani, “Simulation of dynamics and structure formation in high-speed melt spinning” in High Speed fiber spinning, (A. Ziabicki, H. Kawai Eds.), pp.170-200, Wiley-Interscience, New York, 1985

21. R. Menedith, *Textile Progress*, **7**, (1975)
22. M. P. W. Wilson, 1974, “Shrinkage and chain folding in drawn poly(ethylene terephthalate) fibres”, *Polymer*, **15,** 277
23. B.Wunderlich, 1978, “Heat capcities of linear macromolecules”, *Polym Engng Sci*, **18**, 431,
24. H. Shirataki, A. Nakashima, K. Sato, K. Okajima, 1997, “Correlation Between Tensile Properties and Network Draw Ratio for Poly (ethylene terephthalate) Fibers with Wide Range of Molecular Orientation and Crystallinity”, *J Appl Polym Sci,*, **64**, 2631
25. W. O. Statton, J. L. Koenig and M. Hannon, 1970, “Characterization of Chain Folding in poly(Ethylene Terephthalate) Fibers”, *J Appl Phys*, **41**, 4290
26. G.W. Davis and J.R. Talbot, “Polyesters Fibers” in Polymers fibers and Textiles a Compendium, (I. Kroschwitz Eds), pp.680, Wiley-Interscience, New York, 1990
27. K. Yıldırım, Y. Ulcay, O. Kopmaz, 2010, “Forming Regression Based Mathematical Model to Predict PET POY Yarn Properties in Case of Changing Production Parameters”, *Textile Res. J.*, **80**, 411
28. K. Yıldırım, Ş. Altun, M. Kanık, Y. Ulcay, 2009, “Influence of Melt Spinning Conditions on Dye Uptake of Poly(ethylene terephthalate) Fibres”, *Color Technol*, **125**, 151
29. H.C. Karakas, 2004, “A Comparison between the Thermomechanical and Structural Changes in Textured PET Yarns after Superheated Steam and Dry Heat Treatment” *Fibers and Polymers*, **5**, 19
30. K. Yildirim, A.M. Kostem, 002856 number and 02.01.2012 dated technical report, prepared by TUBITAK - BUTAL (Bursa Test and Analysis Laboratory) for Akinbay Tesktil San. ve Tic.A.Ş
31. S. Sardag, O.Ozdemir, M. Kanık 2011, “The Effect of Vacuum Steaming Processes Parameters on Tenacity Properties of Cotton and Viscose Yarns”, *Tekstil ve Konfeksiyon,* **4**, 343