Original article

Textile Research Journal
0(00) 1–11

DOI: 10.1177/0040517511413318
trj.sagepub.com

The effect of heat treatment conditions using the drawing process on the properties of PET filament sewing thread

Andreja Rudolf, Jelka Geršak and Majda Sfiligoj Smole

Abstract

For the production of drawn poly(ethylene terephthalate) (PET) filament threads it is useful to determine the relationship between the drawing process conditions and the resulting product properties. Drawing at an elevated temperature over a defined time usually causes changes of the polymer structural parameters that influence the mechanical properties of the filaments. In order to elucidate the physical background of the drawing process for developing the production process of PET filament sewing thread, the thread was drawn by varying the treatment temperature and contact time. The effect of the treatment conditions on the structural and mechanical properties of the drawn threads was investigated, such as the birefringence, crystallinity, and dynamic mechanical properties of the threads' filaments, as well as the mechanical properties and shrinkage of the treated threads. Using the drawing process, higher temperature and contact time up to the critical values ($T_2 = 220^\circ$C, $n_2 = 9$ turns) change the structural parameters and, consequently, any modifications influence changes in the mechanical properties of the threads. Namely, a higher treatment temperature and contact time increases the degree of crystallinity, the birefringence, and the maxima of the loss tangent and loss modulus temperature, and a significant increase in the breaking tenacity, elastic modulus and the thread's tension at the yield point, and a decrease of the breaking extension is achieved. The observed deterioration of the thread's mechanical properties above the critical treatment conditions is attributed to the destructive phenomena of the supra-molecular structure, resulting in an imperfect structure of the thread's filaments.

Keywords

drawing, heat treatment, mechanical properties, PET filament sewing thread, structural parameters

Introduction

Poly(ethylene terephthalate) (PET) is used for different applications in the forms of films and fibers of different orientation and crystallinity. Orientation and crystallinity in polymers are of great technical importance, since they have the greatest influence on the physical and mechanical properties.[1] Sewing thread as a polymer material displays viscoelastic properties that are conditioned by the supra-molecular structure of the built-in and twisted filaments. During the sewing process, a high tensile and heat load of the thread arises that causes deformation of the thread.[2–11] Deformations are reflected in any structural changes in the thread's twisted fibers and, thereby, changes arise in the mechanical properties.[4] PET multifilament threads for application in the automotive industry have to fulfill high-quality demands regarding strength, elasticity, and friction properties. In order to ensure appropriate properties, a sewing thread undergoes the drawing process at an elevated temperature, in order to improve the thread's mechanical properties. The drawing process is performed between two heated cylinders of a drawing machine. Polyester is usually drawn at temperatures above the polymer glass transition temperature, which

Department of Textile Materials and Design, Faculty of Mechanical Engineering, University of Maribor, Maribor, Slovenia.

Corresponding author:
Andreja Rudolf, Department of Textile Materials and Design, Faculty of Mechanical Engineering, University of Maribor, Smetanova 17, 2000 Maribor, Slovenia
Email: arudolf@uni-mb.si

is imparted by the temperature of the first heated cylinder, whilst the heat treatment of the drawn thread happens on the second heated cylinder. The thread runs around the driven cylinders at different velocities in order to produce the required draw ratio for producing an enhancement in the filaments' structure and, consequently, improving the thread's mechanical properties.

The production of high-modulus and high-strength fibers and yarns has been the subject of intense research over the past few years. It is well known that the important drawing conditions that influence any improvement in PET yarn's structure are the winding speed, temperature, and draw ratio.[12,13] Heat treatment is a time, temperature, and stress-controlled process. When drawing at an elevated temperature, the macromolecular motions are influenced according to the tensile force and heat treatment conditions, thus changing the filaments' mechanical properties.[1,14] In fact, many morphological modifications, such as conformational changes, strain-induced crystallization, and variations in crystal size and distribution, are involved when drawing, and thus orienting, PET.[15,16] Much research work has been done on detailed studies regarding the drawing and heat treatment effect on filaments properties,[17–27] laser-heating zone-drawing and vibrating hot drawing.[28,29]

In earlier research, the structure–properties relationships of the PET filament sewing thread after drawing at an elevated temperature were investigated using different draw ratios (1.05, 1.10, 1.15, 1.20 and 1.25).[30] Dependence was confirmed between the draw ratio, structural parameters, and mechanical properties. A higher draw ratio increases the birefringence, amorphous orientation, and degree of crystallinity, as well as breaking tenacity, elastic modulus, the tension of the thread at the yield point, and decreases the breaking extension. The drawing process was performed at a constant treatment temperature and contact time of the thread with the heated cylinders of the drawing machine. An investigation into differently treated threads by varying the treatment temperature and contact time on the second heated cylinder at a constant draw ratio was performed during this research in order to understand the influence of heat treatment conditions on the thermo-mechanical properties of the PET filament sewing thread.

Thus, much research has focused on determining the effect of drawing and heat treatment conditions on the properties of fibers or yarns, but no studies are currently available regarding the sewing thread. Namely, after the drawing process, two or more yarns are plied and joined by twisting. Then, these threads followed the process of thread dyeing. Both twisting and dyeing processes influence the properties of the thread. Drawing and heat treatment of the thread was performed on a dyed thread in order to achieve the desired properties. Therefore, the main aim of this research was a detailed study of the effect of heat treatment conditions using the drawing process, on the properties of the PET filament sewing thread.

Experimental details

Material and samples preparation

An investigation was performed on a dyed and lubricated PET filament sewing thread, for car industry application, into the influence of thermal treatment conditions during the drawing process on its structural and mechanical properties. The thread was produced from commercial multifilament PET yarn (SANS Fibers) of 28/72/0, i.e. 28 tex, 72 filaments and zero twist; as 3-plied and Z-twisted thread with 210 turns/m and nominal linear density of 84 tex. Furthermore, the thread was dyed with dispersion dye in a dyeing apparatus in the form of packages at 130°C for 45 min. After the dyeing process the thread samples were prepared on an Edmund Erdman drawing machine, type DMW 08/190, by varying the treatment temperature and contact time on the second heated cylinder, whilst the other drawing conditions on the first heated cylinder and draw ratio were constant. The temperature of the first heated cylinder was $T_1 = 120°C$, and number of the thread turns that defines the contact time of the thread with the first heated cylinder was $n_1 = 11$ turns, and the draw ratio $\lambda = 1.14$. During the first treatment procedure the thread was exposed to different treatment temperatures on the second heated cylinder, namely $T_2 = 200$, 210, 220 and 230°C. The contact time of 5 turns was constant. The second treatment involved the preparation of threads by using different contact times on the second heated cylinder, namely $n_2 = 7$, 9, 11 and 13 turns, and the treatment temperature of 200°C was constant. In addition, the sewing threads were surface-treated with a Rayolan HTM lubrication agent.

Analytical methods

Different analytical methods such as calorimetric measurements, birefringence determination, and dynamic mechanical, as well as tensile measurements were used for the investigation into the effect of the drawing process conditions on PET filament sewing thread's properties.

Birefringence. The compensation method according to Ehringhaus was used for filament birefringence determination using an Ortholux Polarization microscope (Leitz-Wetzlar). The diameter of the filament was measured microscopically using an eye-piece micrometer. The measurements were performed at a wavelength of 546.1 nm (white light), and the birefringence (Δn) was calculated from these measurements.[31]

Differential scanning calorimetry. The melting enthalpy was determined by differential scanning calorimetry (DSC) using the DSC-TA-4000-Mettler Toledo measuring system. Each thermogram was recorded from 25–350°C. The heating rate was 10°C/min. The melting enthalpy of the samples (ΔH_{ms}) was determined from the area of the curve's endothermic peak. The degree of crystallinity ($\alpha_{\Delta H}$) was calculated from the melting enthalpy of the samples.[32] The melting enthalpy of the ideal crystalline PET of 140.1 J/g was used for calculation.[33]

Dynamic mechanical analysis (DMA). Measurements of the filaments' dynamic mechanical properties were carried out using a Perkin Elmer DMA7 measuring system at a constant frequency of 10 Hz. The experiment was conducted at 30°C, with a static force of 500 mN and dynamic force of 400 mN. A heating rate of 5°C/min was used. Samples were prepared in the form of a 72 filaments bundle. The β-relaxation transitions (glass transition) were determined by dynamic mechanical analysis, having arisen from the movements of the molecular segments in the amorphous regions. The results were calculated and graphically processed by a program operating at the measuring system. Loss tangent (tanδ) and loss modulus (E'') were determined at the relaxation transitions' maximum, as well as the temperature of the loss modulus ($T_{E''}$).

Shrinkage. Boiling water shrinkage was tested following the standard method ASTM D2102[44] and the initial lengths of the threads were measured. The threads were immersed in boiling water for 30 min. After free shrinkage occurred, the samples were removed from the bath and their lengths were measured. Then, shrinkage (S) was calculated from the initial length (L_0) and the length of the thread after treatment (L_s) and expressed as a percentage.

Mechanical properties. The measurements of the filaments' mechanical properties were carried out on a Vibrodyn 400 dynamometer, connected to a Vibroskop 400 linear density measuring device, according to the standard test method ISO 5079.[34] Breaking tenacity, breaking extension and elastic modulus at 1% extension were calculated from the obtained results. Measurements were carried out under standard conditions.[35]

The mechanical properties of the yarn and drawn threads were measured on an Instron 6022 dynamometer according to the standard test method ISO 2062[36] under standard conditions.[36] A mean stress–strain curve was constructed from the measurements, and the visco-elastic parameters were calculated and determined from the mean curve,[37] and from the first, second and third derivative of the experimental curves (σ', σ'', σ'''), respectively. The elastic modulus (E_0) was determined at the gradation points of the first derivative curve, where the second derivative curve intersects the *x*-axis. The tension at the yield point (σ_y) was determined on the curve $\sigma(\varepsilon)$ at the position of $\sigma'''(\varepsilon) = 0$. The linear density of the threads was determined according to the standard test method ISO 1889.[38]

Results and discussion

Influence of the treatment temperature and contact time on the structural parameters

The structural parameters of the drawn PET filament thread conditioned by the heat treatment temperature on the second heated cylinder at a constant contact time of 5 turns are presented in Table 1. The influence of the heat treatment temperature on the structural parameters is presented in Figure 1.

The degree of crystallinity of 0.390, determined from the DSC curve, and a birefringence of 0.2127 were found for the untreated yarn's filament. An increase in the degree of crystallinity was found for the heat treated threads when compared with the untreated yarn, Table 1. In Figure 1(a) a trend of increase in the degree of crystallinity was observed when increasing the treatment temperature up to 220°C. The highest degree of crystallinity 0.437 was achieved at a treatment temperature of 220°C. The heat treatment increases molecular orientation, as confirmed by the measurements of the filament's birefringence (Table 1) when compared with the untreated yarn. The trend of increase in the birefringence with increasing the treatment temperature was only observed up to a treatment temperature of 220°C, where the highest Δn of 0.2350 was achieved (Figure 1(a)). A similar effect from a heat-setting temperature on increased crystallinity and molecular orientation for taut annealed multifilament PET yarn was observed by Gupta and Kumar.[20] They observed an increase in the crystallinity and crystallite size, as well as in birefringence when the heat-setting temperature increases. Bigger crystals can grow by merging the small crystallites into bigger ones, and during this process some or most of the small crystals dissolve, depending on the temperature of the heat setting. In addition, the perfection of the crystallites could be improved by the disappearance of the crystal defects.

Table 1. Structural parameters of the drawn PET filament threads conditioned by the heat treatment temperature on the second heated cylinder

Heat treatment conditions		Sample ID	Degree of crystallinity $\alpha_{\Delta H}$	Birefringence Δn	Loss tangent $\tan\delta_{max}$	Loss modulus E''_{max} (10^6 Pa)	Temperature of loss modulus $T_{E''max}$ (°C)	Shrinkage S (%)
T_2 (°C)	n_2 (turns)							
200	5	D_1	/	0.2237	0.154	4.31	/	2.55
210		D_2	0.421	0.2268	0.165	4.65	127.55	3.05
220		D_3	0.437	0.2350	0.151	3.37	128.07	4.55
230		D_4	0.432	0.2340	0.145	5.50	127.89	2.27

Furthermore, Huisman and Heuvel[12] confirmed an increase of crystallinity, and both molecular and crystalline orientation, with increasing treatment temperature. They found that heat treatment at low temperature results in many small crystals, whilst at a high treatment temperature slightly smaller-sized crystallites were formed in the PET yarn. On this basis, it could be supposed that with any increase in the treatment temperature up to 220°C, the heat input in the PET sewing thread increases and the mobility of the macromolecular chains is enhanced. As the temperature increases, the mobility of the polymer segments increases. This allows easier alignment of the polymer segments to the crystals. In addition, increased temperature and tensile deformation by the drawing process probably causes straightening and sliding of the strained intrafibrillar tie molecules, which proceed to the crystalline domains. This is confirmed by an increased degree of crystallinity and birefringence after the heat treatment process on the drawing machine. However, a decrease in the degree of crystallinity and birefringence was observed at the highest treatment temperature of 230°C (Table 1). We suppose that the observed structural changes arise from variations in the non-crystalline regions and at the beginning of the small crystallites' melting process.

Changes in the physical state were observed when scanning the temperature during the DMA experiments, and the loss tangent (tgδ) and loss modulus (E'') were determined at the relaxation transitions' maxima, as well as the temperature of the loss modulus ($T_{E''}$), which is used for interpretation of the glass transition temperature.[25] The filaments' loss modulus E'' represents the molecular relaxation processes in the non-crystalline regions of the polymer, and is a function of the amount and degree of orientation of the non-crystalline chains in the sample.[25] With any increase in amorphous orientation there is an increase in resistance to thermal mobility; consequently, a lower loss modulus appears and the glass transition temperature is higher.[25] Therefore, we used the loss modulus maximum as a criterion for an estimation of the amorphous orientation. The β-relaxation transitions maxima (glass transition) of the untreated yarn's filaments were: loss tangent 0.131, loss modulus 4.48×10^6 Pa, whilst the temperature of the loss modulus was 124.21°C. When compared with the untreated yarn with heat-treated threads on the second heated cylinder, an increase in the loss tangent and the temperature of the loss modulus maximum up to 220°C was achieved,

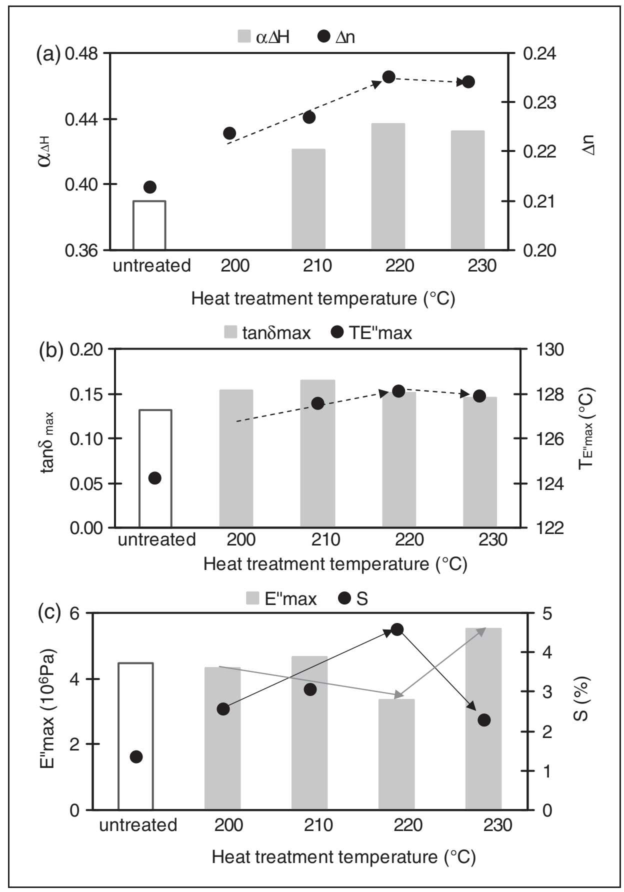

Figure 1. Influence of the heat treatment temperature on structural parameters. Threads were treated at constant contact time of 5 turns on the second heated cylinder. (a) degree of crystallinity/birefringence, (b) loss tangent/temperature of the loss modulus, (c) loss modulus/shrinkage.

whilst the loss modulus decreased (Table 1). A trend of $T_{E''max}$ and glass transition temperature increase (Figure 1(b)), respectively, and a decrease in the E''_{max} with increasing treatment temperature up to 220°C was observed (Figure 1(c)). With an increase of the treatment temperature to 230°C, a decrease of the glass transition temperature and an increase in the loss modulus was achieved, as can be seen in Table 1. The heat treatment increased the loss tangent (Table 1), whilst any significant dependence on increasing temperature was not observed. It achieved the highest value at the treatment temperature of 210°C. On the basis of the DMA results, it could be concluded that increasing the treatment temperature up to 220°C on the second heated cylinder improves the amorphous orientation. Because the heat treatment was performed just after the drawing process on a drawing machine, the sewing thread was heated in a taut state. Therefore, the tensile deformation and increasing temperature up to 220°C contributes to greater alignment of the macromolecules along the filament's axis, and probably increases trans conformation in the non-crystalline macromolecules. The increase in the amorphous orientation induced by the heat treatment also contributes to the increase in the loss tangent and glass transition temperature, and to the loss modulus decrease.

The results of this investigation are confirmed by the results obtained by Murayama.[14] An increase in the degree of crystallinity and molecular orientation was found when increasing the treatment temperature and drawing the PET yarn. As the crystallinity and molecular orientation increased the E''_{max} decreased, and a shift of E''_{max} to higher temperatures was evident, whilst an increase in the width of the E'' peak is attributed to an increased range of order.[14] An increase of the amorphous orientation with any increasing heat-setting temperatures of the samples treated in the taut state was also found by Gupta and Kumar.[20] On the other hand, they determined a decrease in the amorphous orientation with increasing temperature for free-annealed samples. Increase in non-crystalline orientation with increasing treatment temperature is also confirmed by the results of the shrinkage of the threads in boiling water. The thermally induced shrinkage occurs as a result of disorientation in the oriented non-crystalline regions of the fibers.[39] It has been shown that, in PET yarns, shrinkage is a unique function of the product of the amorphous volume fraction and amorphous-orientation factor.[13] It was observed that taut annealed samples show a higher shrinkage than the free-annealed samples, as a result of the contraction of highly extended interfibrillar tie molecules.[40] Therefore, the higher orientation of the non-crystalline regions is the reason for higher fiber shrinkage caused by a disorientation process occurring in the oriented amorphous phase. The shrinkage was 1.32% for the untreated yarn. The shrinkage increased during heat treatment (Table 1). A trend towards an increase in the shrinkage was observed when increasing treatment temperature up to 220°C (Figure 1(c)), where shrinkage of 4.55% was achieved. When heat treatment at a temperature of 230°C was performed, a decrease in shrinkage to the value of 2.27% occurred. In addition, it was found that heat treatment on the second cylinder at a temperature 230°C leads to decreases in the birefringence, degree of crystallinity, amorphous orientation, and glass transition temperature. Therefore, a significant decrease in shrinkage is reasonable, whilst the destruction of the filaments' supra-molecular structure begins initially in the non-crystalline phase and proceeds to the crystalline domains.

The structural parameters of the drawn PET filament thread, conditioned by the heat treatment contact time on the second heated cylinder at the constant temperature of 200°C, are collected in Table 2. The influence of the heat treatment contact time on the structural parameters is presented in Figure 2.

The heat treatment and increasing contact time of the thread around the second cylinder improves the structural parameters when compared with the untreated yarn, as can be seen in Figure 2. With an increase in the contact time up to 9 turns, a trend was found towards an increase in the degree of crystallinity, birefringence, loss tangent, and glass transition temperature (Figure 2). Any significant influence of the contact time was not observed within the range between 7 and 9 turns, on the loss modulus and shrinkage. Both parameters express high amorphous orientation obtained by heat treatment, which is independent of the contact time. With regard to the findings of Van den Heuvel et al.,[26] Ajji et al.[15] and Suzuki and Mochiduki,[29] we are assuming that increasing the heating time up to 9 turns increases the trans conformation in the non-crystalline molecules. Macromolecules of the taut thread's filaments above the glass transition temperature tend to arrange along the filament's axis. Therefore, the amount of gauche conformations diminishes and proceeds to trans conformations. In addition, a high constant treatment temperature also contributes to an increase in the degree of crystallinity and both crystalline and amorphous orientation, as well as to an increase in the glass transition temperature.

There are some structural changes that are unexpected. These are the low value of the loss tangent at a contact of 7 turns, and the shrinkage obtained at a contact time of 9 turns. Moreover, when comparing the threads marked D1, E1 and E2 that were heat treated at 200°C at different contact times (5, 7 and 9 turns), any increase in the birefringence and amorphous

Table 2. Structural parameters of the drawn PET filament threads conditioned by the heat treatment contact time on the second heated cylinder

Heat treatment conditions		Sample ID	Degree of crystallinity $\alpha_{\Delta H}$	Birefringence Δn	Loss tangent $\tan\delta_{max}$	Loss modulus E''_{max} (10^6 Pa)	Temperature of loss modulus $T_{E''max}$ (°C)	Shrinkage S (%)
T_2 (°C)	n_2 (turns)							
200	7	E_1	0.448	0.2310	0.129	3.85	128.94	4.16
	9	E_2	0.461	0.2351	0.151	3.45	129.75	4.08
	11	E_3	0.449	0.2224	0.150	4.49	124.93	1.03
	13	E_4	0.439	0.2236	0.125	4.75	124.69	0.77

orientation with increasing treatment time can be seen in Tables 1 and 2. The unexpected structural changes caused under heat treatment conditions are attributed to the non-uniform treatment of the filaments due to their different positions in the twisted thread's cross-sections. Namely, sewing thread has a geometry of parallel filaments that are twisted to achieve sufficient cohesiveness. In this form the filaments are inclined under defined twist angles in regard to the longitudinal thread axis. According to idealized helical yarn geometry, the sewing thread is assumed to be circular in cross-section and composed of a series of concentric cylinders of differing radii, where the filaments follow a helical path around one of the concentric cylinders.[41] Therefore, it is supposed that only a part of the thread's filaments are in direct contact with the heating cylinder. On the other hand, it should be stated that textile fibers are generally non-homogeneous[42] and, therefore, property variations along the fiber axis can usually be observed. Furthermore, when prolongation of the contact time within the range between 11 and 13 turns of the thread around the second heated cylinder at a constant temperature of 200°C was performed, a trend was observed towards a decrease in the degree of crystallinity, birefringence, loss tangent, glass transition temperature, and amorphous orientation (Figure 2). A significant decrease in non-crystalline orientation is especially evident from the threads' shrinkage. Namely, at a contact time of 9 turns of the thread around the second heated cylinder, a shrinkage of 4.08% was found, whilst at 13 turns a significant decrease to a value of 0.77% was achieved (Table 2). Prolongation of the treatment time enables longer heat transfer into the thread, which is in direct contact with the heated cylinder. This leads to a significant mobility of the macromolecules. An observed decrease in amorphous orientation could arise from conformational variations in non-crystalline regions. On the basis of the gained results, we conclude that at heat treatment contact time over more than 9 turns, the rearrangements in the amorphous layers affect the crystallites' outer layers regions, resulting in a slight destruction of the supramolecular structure.

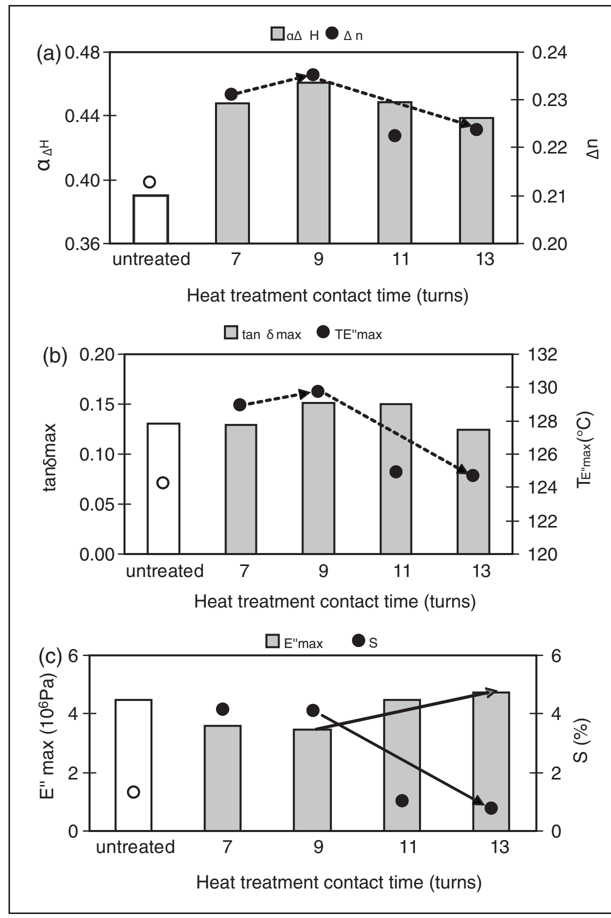

Figure 2. Influence of the heat treatment contact time on structural parameters. Threads were treated at constant temperature of 200°C on the second heated cylinder. (a) degree of crystallinity/birefringence, (b) loss tangent/temperature of the loss modulus, (c) loss modulus/shrinkage.

It could be concluded that heat treatment by the drawing process improves the structural parameters of the PET threads' filaments. Increasing the treatment temperature within the range between 200°C and 220°C, and increasing the contact time within the range between 7 and 9 turns of the thread around the

Table 3. Mechanical properties of the drawn PET filament threads conditioned by the heat treatment temperature on the second heated cylinder

Heat treatment conditions		Sample ID	Breaking tenacity σ_b (cN/tex)		Breaking extension ε_b (%)		Elastic modulus E_0 (cN/tex)		Tension of the thread at yield point σ_y (cN/tex)
T_2 (°C)	n_2 (turns)		Filament	Thread	Filament	Thread	Filament	Thread	
200	5	D_1	69.4	58.49	18.9	16.72	394	4.75	9.29
210		D_2	68.2	58.12	16.3	15.93	411	6.00	8.66
220		D_3	71.4	60.72	14.1	12.13	463	7.41	9.27
230		D_4	69.2	59.24	17.1	17.86	391	5.21	8.52

second heated cylinder, a positive trend was observed towards an increase in the degree of crystallinity, birefringence, amorphous orientation, and glass transition temperature. It should be emphasized that the drawing process at a constant draw ratio and elevated temperature (first cylinder) is performed just before the additional heat treatment on the second cylinder, therefore this drawing condition also attributes to improved filaments properties. The process of heat treatment on the second cylinder at a temperature above 220°C and above the contact time of 9 turns, leads to a decrease in the birefringence, degree of crystallinity, amorphous orientation, and glass transition temperature. We presume that the observed structural changes arise from conformational variations in the non-crystalline regions, and an initiation of the small crystallites' melting process.

Influence of the treatment temperature and contact time on the mechanical properties

The mechanical properties of the drawn PET multifilament threads conditioned by the heat treatment temperature are presented in Table 3. The influence of the heat treatment temperature on the mechanical properties of the threads is shown in Figure 3.

When heat treatment of the PET filament sewing thread is performed, the modification of the filaments' structure affects any changes in the thread's mechanical properties. The mechanical properties for the untreated yarn's filament were: breaking tenacity 70.80 cN/tex, breaking extension 17.6%, and elastic modulus 548 cN/tex, whilst the mechanical properties for the untreated yarn were: breaking tenacity 62.09 cN/tex, breaking extension 13.54%, elastic modulus 7.60 cN/tex, and the tension of the yarn at the yield point 11.62 cN/tex. Increasing the temperature of the second cylinder influences changes in the mechanical properties of the filaments and threads, as shown in Figure 3. After heat treatment at the lowest temperature of 200°C the thread, when compared with the untreated yarn, had lower values for breaking tenacity,

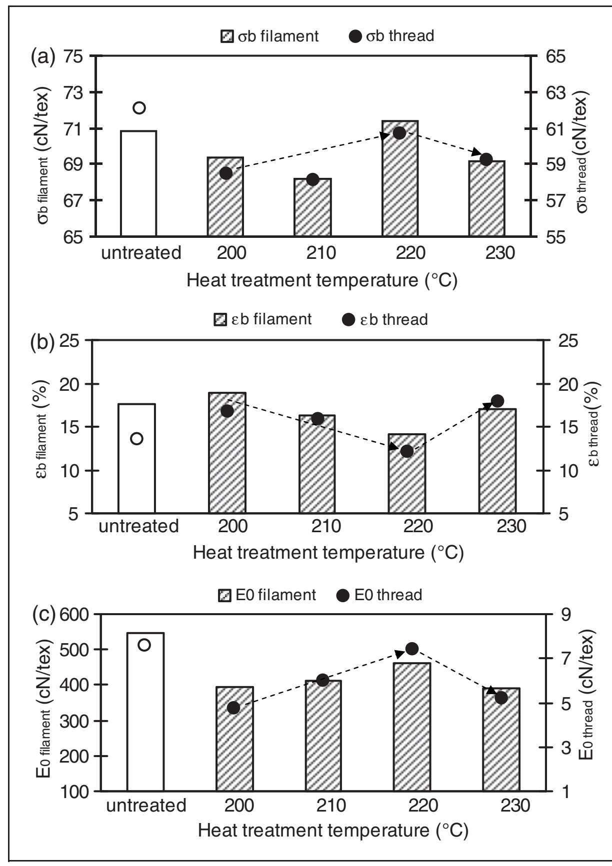

Figure 3. Influence of the heat treatment temperature on mechanical properties. Threads were treated at constant contact time of 5 turns on the second heated cylinder. (a) breaking tenacity, (b) breaking extension, (c) elastic modulus.

elastic modulus, and the tension at the yield point, and a higher value for breaking extension (Table 2). This phenomenon arises due to the twisting of the thread, where the thread shapes into a spiral form. Within this form, the filaments are inclined at a defined angle in regard to the thread axis, which decreases the tensile strength and increases the breaking extension.[41,43] Furthermore, the torsion and compressive loadings of

Table 4. Mechanical properties of the drawn PET filament threads conditioned by the heat treatment contact time on the second heated cylinder

Heat treatment conditions			Breaking tenacity σ_b (cN/tex)		Breaking extension ε_b (%)		Elastic modulus E_0 (cN/tex)		Tension of the thread at yield point σ_y (cN/tex)
T_2 (°C)	n_2 (turns)	Sample ID	Filament	Thread	Filament	Thread	Filament	Thread	
200	7	E_1	70.0	62.04	12.3	11.99	482	7.70	10.22
	9	E_2	73.1	61.59	12.7	12.12	490	7.40	8.62
	11	E_3	67.0	59.26	18.8	16.70	378	5.80	7.69
	13	E_4	67.4	59.63	16.9	17.95	379	5.25	7.77

the thread-twisted filaments originate when twisting, and in particular decrease the elastic modulus. When increasing the treatment temperature up to 220°C a trend towards an increase in the breaking tenacity, and especially the elastic modulus is observed, whilst the breaking extension decreases with increasing treatment temperature (Figure 3). The breaking tenacity of the filament achieved the highest value of 71.40 cN/tex, whilst the highest value for the thread's breaking tenacity was 60.72 cN/tex. When a treatment temperature of 220°C was used, the breaking extension of the filament was 14.10% and for the thread 12.13%, whilst the elastic modulus of the filament was 463 cN/tex and for the thread 7.41 cN/tex, respectively. The tension of the thread at the yield point does not show any tendency to vary with increasing treatment temperature, as can be seen in Table 3. It achieves its highest value of 9.29 cN/tex at a treatment temperature of 200°C. By increasing the treatment temperature, the most extensive change was achieved for the breaking extension and elastic modulus when compared with the breaking tenacity (Table 3). It is well known that improved mechanical properties are attributed to higher crystallinity, and to higher orientation of the macromolecules in both the crystalline and amorphous phases. Furthermore, Gupta and Kumar[22] found, in regard to the taut heat-treated samples, an increase in the breaking tenacity and tension at the yield point with increasing temperature, and a decrease in the breaking extension. They established that, beside the crystalline orientation and the sizes and distribution of the crystallites, the amount and orientation of the amorphous phase influences the mechanical properties.[21,22] The results of this investigation coincide with their findings. Deterioration in the mechanical properties above the treatment temperature of 220°C coincides with the observed structural changes in the presented research.

The mechanical properties of the drawn PET multifilament threads conditioned by the heat treatment contact time are presented in Table 4. The influence of the heat treatment contact time on the mechanical properties of the threads is shown in Figure 4.

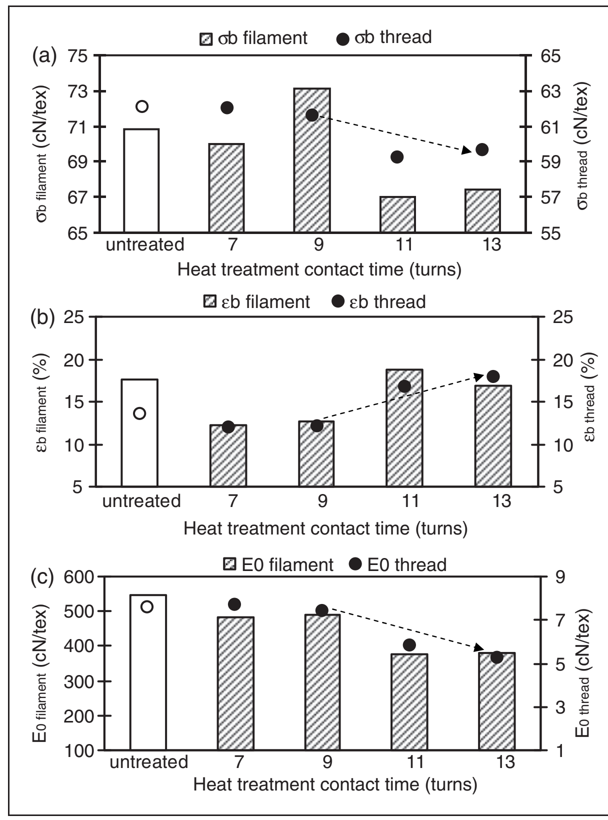

Figure 4. Influence of the heat treatment contact time on mechanical properties. Threads were treated at constant temperature of 200°C on the second heated cylinder. (a) breaking tenacity, (b) breaking extension, (c) elastic modulus.

After the treatment at the lowest contact time of the thread with the second heated cylinder, the thread, when compared with the untreated yarn, had similar values for breaking tenacity and elastic modulus, and a significant decrease in the breaking extension was achieved, as can be seen in Figure 4. This means that even a low contact time has a great influence on changes in the thread's mechanical properties. Furthermore, any increase in the contact time of the thread around the second heated cylinder up to the critical value of 9 turns forms a better fibrillar structure, which is confirmed with increased degree of crystallinity, birefringence, amorphous orientation, and glass transition temperature. Thereby, an increase in the breaking tenacity and elastic modulus was achieved, whilst the breaking extension decreased. A similar influence of the treatment time, especially on amorphous orientation, breaking extension and elastic modulus, can be seen in Figures 2 and 4. The tension of the thread does not show any expected tendency towards variation with increasing heat treatment contact time. It achieved the highest value of 10.22 cN/tex at a contact time of 7 turns. By increasing the treatment time above 9 turns, a decrease in the breaking tenacity, elastic modulus, and the tension of the thread at the yield point was achieved, and a significant increase in the breaking extension arose, as shown in Figure 4. With prolonged treatment time there is a relationship between the structural and mechanical properties. It can be concluded that increasing the treatment contact time up to 9 turns improves the tensile properties of the threads. The breaking tenacity of the filament achieves the highest value of 73.10 cN/tex, whilst the highest value of the thread breaking tenacity is 62.04 cN/tex. When a contact time of 9 turns was used, the breaking extension of the filament was 12.70% and of the thread 12.12%, whilst the elastic modulus of the filament was 490 cN/tex and that of the thread 7.40 cN/tex, respectively. Increasing the treatment time of the PET filament sewing thread on the second heated cylinder has a greater influence on changes in the breaking extension and elastic modulus, when compared with the breaking tenacity (Table 2).

On the basis of this research it could be concluded that the heat treatment process leads to a more perfect fibrillar structure of the threads' filaments, thereby an increase in the breaking tenacity, elastic modulus, and the tension of the thread at the yield point, and a decrease in the breaking extension can be achieved up to a critical treatment temperature of 220°C and treatment contact time of 9 turns. The deterioration of the mechanical properties due to the heat treatment process at temperatures above 220°C and contact time above 9 turns was found, and coincides with the observed structural parameters. The breaking tenacity, elastic modulus, and the tension of the thread at the yield point were decreased, whilst an increase in the breaking extension was achieved.

Conclusion

The effect of the heat treatment conditions during the drawing process on the structural and mechanical properties of PET filament sewing thread was investigated in this paper. The trend towards interdependence was confirmed between the heat treatment conditions and structural parameters within the range between a treatment temperature of 200°C to 220°C, and a contact time of 7–9 turns of the thread around the second heated cylinder. Increasing the treatment temperature and contact time increases the degree of crystallinity, birefringence, and the maxima of the loss tangent and loss modulus temperature. These structural modifications also comprise changes in the thread's mechanical properties, i.e. an increase in the breaking tenacity, elastic modulus, and the tension of the thread at the yield point, and a decrease in the breaking extension is significant when the heat treatment temperature increases up to 220°C, and when the contact time increases up to 9 turns. Deterioration of the thread's mechanical properties above a temperature of 220°C and a contact time above 9 turns of the thread around the second heated cylinder is attributed to the beginning of supramolecular structure destruction, resulting in a less than perfect structure of the thread's filaments.

Funding

This work was supported by the Slovenian Research Agency (grant number 4010-945).

Conflict of interest

The authors declare that they have no conflicts of interest.

References

1. Ward IM and Hadley DW. An introduction to the mechanical properties of solid polymers. Chichester, England: John Wiley and Sons, 1995.
2. Rudolf A and Geršak J. Influence of twist on alterations in fibers' mechanical properties. *Textile Res J* 2006; 76: 134–144.
3. Rudolf A and Geršak J. Influence of sewing speed on the changes of mechanical properties of differently twisted and lubricated threads during the process of sewing. *Tekstil* 2007; 56: 271–285.
4. Rudolf A, Geršak J, Ujhelyiova A and Sfiligoj Smole M. Study of PES sewing thread properties. *Fibers Polym* 2007; 8: 212–217.
5. Liasi E, Du R, Simon D, Dimitrejevic JB and Liburdi F. Heating of an industrial sewing machine needle. *Int J Cloth Sci Technol* 1999; 11: 300–314.
6. Li Q, Liasi E, Simon D and Du R. A study on the needle heating in heavy industrial sewing-Part 1: analytical models. *Int J Cloth Sci Technol* 2001; 13: 87–105.
7. Midha VK, Mukhopadhyay A, Chatopadhyay R and Kothari VK. effect of process and machine parameters on changes in tensile properties of threads during high-speed industrial sewing. *Textile Res J* 2010; 80: 491–507.

8. Midha VK, Mukhopadhyay A, Chatopadhyay R and Kothari VK. studies on the changes in tensile properties of sewing thread at different sewing stages. *Textile Res J* 2009; 79: 1155–1167.
9. Geršak J. Einfluss der technologischen Parameter auf die Nähnadeltemperatur im Nähprozess. *TPI Text-Prax Int* 1986; 41: 280–286.
10. Geršak J. Untersuchungen über die Größe der Belastung des Nähfadens während des Stichbildungsprozesses. *Bekleid + Wäsche* 1988; 40: 37–41.
11. Geršak J. Reduction in thread strength as a cause of loading in the sewing process. *Int J Cloth Sci Technol* 1991; 3: 6–12.
12. Huisman R and Heuvel HM. A quantitative description of the effect of process conditions on the physical structure of poly ethylene terephthalate yarns with an application to dyeing behavior. *J Appl Polym Sci* 1978; 22: 943–965.
13. Huisman R and Heuvel HM. The effect of spinning speed and drawing temperature on structure and properties of poly ethylene terephthalate yarns. *J Appl Polym Sci* 1989; 37: 595–616.
14. Murayama T. Dynamic mechanical analysis of polymeric material. Amsterdam, Oxford, New York: Elsevier Scientific Publishing Company, 1978.
15. Ajji A, Cole KC, Dumoulin MM and Brisson J. Amorphous orientation of poly ethylene terephthalate by X-ray diffraction in combination with Fourier transform infra-red spectroscopy. *Polymer* 1995; 36: 4023–4030.
16. Ajji A, Guèvremont J, Cole KC and Dumoulin MM. Orientation and structure of drawn poly ethylene terephthalate. *Polymer* 1996; 37: 3707–3714.
17. Cullerton DL, Ellison MS and Aspland JR. Effect of commercial heat setting on the structure and properties of polyester carpet yarn. *Textile Res J* 1990; 50: 594–606.
18. Elenga R, Seguela R and Rietsch F. Thermal and mechanical behaviour of crystalline polyetilene terephthalate: effects of high temperature annealing and tensile drawing. *Polymer* 1991; 32: 1975–1982.
19. Göschel U and Urban G. Supermolecular structure of oriented and semicrystalline poly ethylene terephthalate as revealed by the electron density correlation function from small-angle X-ray scattering studies. *Polymer* 1995; 36: 3633–3639.
20. Gupta VB and Kumar S. The effect of heat setting on the structure and mechanical properties of poly ethylene terephtalate fibre, I. Structural changes. *J Appl Polym Sci* 1981; 26: 1865–1876.
21. Gupta VB and Kumar S. The effect of heat setting on the structure and mechanical properties of poly ethylene terephthalate fiber, II. The elastic modulus and its dependence on structure. *J Appl Polym Sci* 1981; 26: 1877–1897.
22. Gupta VB and Kumar S. The effect of heat setting on the structure and mechanical properties of poly ethylene terephthalate fiber, IV. Tensile properties other than modulus and their dependence on structure. *J Appl Polym Sci* 1981; 26: 1897–1905.
23. Pal SK, Ghandhi RS and Kothari VK. Influence of texturing parameters on structural properties of microfiber polyester yarns. *Chem Fibers Int* 1995; 45: 418–420.
24. Peterlin A. Morphology and properties of crystalline polymers with fiber structure. *Textile Res J* 1972; 42: 20–30.
25. Samuels RJ. Quantitative structural characterization of the mechanical properties of poly ethylene terephthalate. *J Polym Sci: Part 2* 1972; 10: 781–810.
26. Van Den Heuvel CJM, Heuvel HM, Faassen WA, Veurink J and Lucas LJ. Molecular changes of PET yarns during stretching measured with rheo-optical infrared spectroscopy and other techniques. *J Appl Polym Sci* 1993; 49: 925–934.
27. Sfiligoj Smole M and Zipper P. WAXS analysis of structural changes of poly ethylene therephthalate fibers induced by supercritical-fluid dyeing. *Colloid Polym Sci* 1998; 276: 144–151.
28. Kunugi T and Suzuki A. Preparation of high-modulus polyethylene terephthalate fibers by vibrating hot drawing. *J Appl Polym Sci* 1996; 62: 713–719.
29. Suzuki A and Mochiduki N. Mechanical properties and superstructure of polyethylene terephthalate fibers zone-drawn and zone-annealed by CO_2 laser heating. *J Appl Polym Sci* 2001; 82: 2775–2783.
30. Rudolf A and Sfiligoj-Smole M. Structure–properties relations of the drawn polyethylene terephthalate filament sewing thread. *J Appl Polym Sci* 2008; 110: 2641–2648.
31. Nettelnsroth K. *Melliand Textilberichte* 1972; 63: 212.
32. Widmann G and Riesen R. *Thermal Analysis*. Heidelberg: Dr. Alfred Hüthing Verlag, 1987.
33. Wunderlich B. *Macromolecular Physics, Volume 3*. Crystal Melting. New York: Academic Press, 1980.
34. ISO 5079-1977. *Textiles – Man-made fibres – Determination of breaking strength and elongation of individual fibres*. International Organization for Standardization, 1977.
35. ISO 139-1973. *Textiles – Standard atmospheres for conditioning and testing*, 1st ed, International Organization for Standardization, 1973.
36. ISO 2062-1972. *Textiles – Yarn from packages – Method for determination of breaking load and elongation at the breaking load of single strands – CRL, CRE and CRT testers*, 1st ed, International Organization for Standardization, 1972.
37. Bukošek V. Računalniško vrednotenje viskoelastičnih lastnosti vlaken in Slovene. *Tekstilec* 1983; 26: 24–29.
38. ISO 1889-1987. *Textiles- Determination of linear density of single and plied yarns reel method*. International Organization for Standardization, 1987.
39. Lewin M and Pearce EM. *Fiber Chemistry, Handbook of Fiber Science and Technology: Volume IV*. New York and Basel: Marcel Dekker Inc, 1985.
40. Gupta VB. The nature of coupling between the crystalline and amorphous phases and its effect on the properties of heat-set polyethylene terephthalate fibres. *J Text Inst* 1995; 86: 299–317.

41. Hearle JWS, Grosberg P and Backer S. *Structural Mechanics of Fibers, Yarns and Fabrics, Volume 1*. USA: Wiley-Interscience, 1969.
42. Gregor-Svetec D and Sfiligoj-Smole M. Homogeneity of polypropylene and polyethyleneterephtalate fibers. *Textile Res J* 2010; 80:1569–1580.
43. Eirich FR. *Rheology, Theory and Applications: The Geometry of Tensional Behaviour of Yarns*. Brooklyn, New York: Polytechnic Institute of Brooklyn, 1969.
44. ASTM D2102-96. *Standard Test Method for Shrinkage of Textile Fiber Bundle Test*. ASTM International, 1996.