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Dependence of Structural Characteristics of Polyamide Textured Yarns on the Parameters of the False Twist Yarn Texturing Process

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Abstract
In the false twist texturing process, due to the action of mechanical forces and heat, a disorientation of structural elements happens at all levels of the supramolecular structure. These changes are related to changes in the texturing parameters and mechanical properties of yarn. In this paper, investigated is the effect of technical-technological texturing parameters in the false twist texturing process on the structure of PA6.6 yarns. POY multifilament PA6.6 with a fineness of 22f07x1 dtex was used as experimental material. The yarn was textured on a friction texturing machine – ICBT model FT 15 E3. The exiting yarn speed (V_1) changed as 600, 700, 800 and 900 m/min; the heater temperature (T) was 200, 210 and 220°C, and the ratio of the disk surface speed to the linear yarn speed (D/Y) was 1.9 and 2.1. The values of strain were kept constant at 1.305 (tension in texturing zone) and 0,954 in the winding zone. Analysed were the density, degree of crystallinity, degree of orientation, single filament diameter, the content of -NH_2 and -COOH end groups, and the total content of end groups. From the results obtained it can be seen that the effect of the heater temperature is more significant than that of V_1 and D/Y on the structural characteristics analysed. Analysing the experimental results it was found that linear positive correlations were established between the texturing speed and end (-NH_2) groups, the texturing speed and end (-COOH) groups and the texturing speed and the total content of end groups. The correlation factor between the process parameters and yarn structural characteristics analysed is determined.

Key words: *false twist, structural characteristics, textured yarns.*

Introduction

Polyamide fibers are thermoplastic materials that can be reshaped by heating, and after cooling they retain the shape obtained in a plastic state. Conditionally, by reheating it is possible to reshape them again and set them. In the texturing process, structural changes occur in fiber polymer, primarily the disorientation of macromolecule chains [1-2]. On the one hand, torque tension affects the disorientation of molecule chains, and on the other hand the tensile load leads to further orientation of crystal and amorphous regions. Increasing the heater temperature causes an increase in the textured yarn degree of crystallinity [3], while the strength and tensile modulus decrease due to the decrease in the crystal orientation degree. The influence of molecule orientation is higher on mechanical properties of yarn than that of the crystal region increase.

The fiber degree of crystallinity and orientation represent significant parameters of the fiber supramolecular structure. The ratio of contribution of some characteristics of crystal (or amorphous) regions to the same characteristics of both regions represents the degree of crystallinity [4, 5]. It is related to both the polymer production and processing as well as the processing of polymer products. Some of the suitable testing methods are as follows: electron microscopy in polarised light, x-ray diffraction, nuclear magnetic resonance, infrared spectroscopy, density measurement, melting heat measurement, etc.

The second important characteristic of the supramolecular structure is the orientation of molecules in a direction expressed by the orientation degree. It can be considered as the orientation of crystal regions and amorphous regions as well as the average orientation, which is the mean value of the previous two types. The orientation of molecules in crystal region can be expressed in regard to fiber axis, to some crystalline direction (usually axis c), or to some external direction serving as calibration. The degree of orientation in the crystal region can be determined by x-ray diffraction, while the method based on measuring birefringence gives the average orientation prevailing in both regions. As a parameter of the supramolecular structure, the degree of orientation has a high influence on the breaking strength, elongation at break [6, 7] and other mechanical properties. When the orientation of molecules in the fiber is lower, its elongation at break is higher and vice versa.

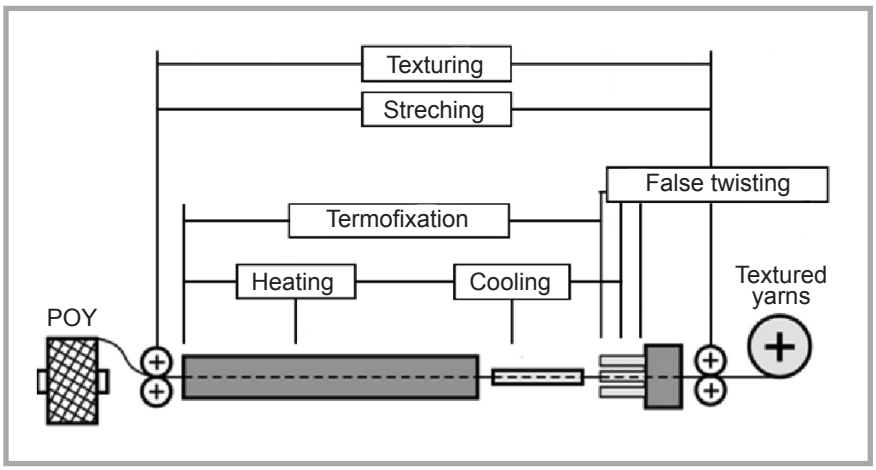

Figure 1. *Scheme of the friction texturing process.*

Experimental

Materials

PA6.6 multifilament yarn with a fineness of 22f07 x 1 dtex, TWD Germany, was used as experimental material. Partial oriented (POY) filament yarn was textured under industrial conditions on a friction texturing machine, model FT 15 E3 (with long heater), ICBT France. Variable technical – technological parameters were as follows: temperature in the heating zone: 200, 210 & 220 °C, exiting textured yarn speed: 600, 700, 800 & 900 m/min, disk surface speed to linear yarn speed ratio (D/Y): 1.9 and 2.1. For the ratio D/Y 1.9 the disk surface speed ranges from 1140 m/min to 1710 m/min, depending on the input yarn speed in the friction unit, which set on the computer of the machine. For the ratio D/Y 2.1 the disk surface speed ranges from 1260 m/min to 1890 m/min. Tension values were held constant in the texturing zone (stretching) as 1.305 and in the winding zone as 0.954. The disk configuration was 1-4-1, a disk type ceramic with a diameter of 52 mm and thickness of 9 mm. A scheme of the friction texturing process with the most important technical – technological parameters is shown in ***Figure 1***.

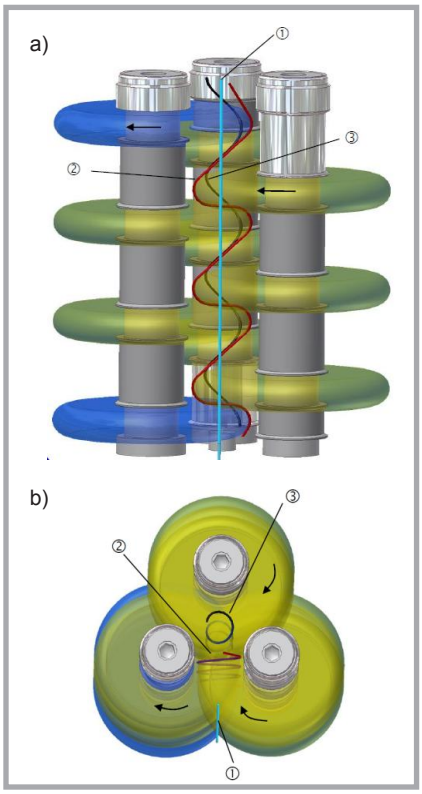

Figure 2. *a) Yarn path through the friction unit and b) the arrangement of twisting disks.*

Figure 2.a and ***2.b*** shows the yarn path through the friction unit as well as the arrangement, direction and surface disk speed for twisting.

Test methods

The following test methods were used for testing the laboratory material:

- The fiber degree of cristallinity was determined by the specific mass test method, which is based on the fact that crystal regions have a thicker package due to the order and more regular geometric arrangement of molecules, and therefore they have a higher density compared to amorphous regions. To use this method, it is necessary to know the density previously. Using calibration curves for PA6.6 fibers, the density is correlated to the degree of crystallinity.
- The method for determination of the density (specific mass) was used for determination of the specific mass. Prepared bundles of PA6.6 fibers were poured over with 20 ml of benzene (C_6H_6), MERCK, Germany. After removing air bubbles, by pressing the fibers on the vessel wall, carbon tetrachloride (CCl_4), HEMOS, Serbia, is added slowly, until fibers start to float in this solvent mixture. In that moment the density of the testing sample is equal to thatof the mixture.
- For determination of the fiber degree of orientation, an interferential method based on polarising microscopy and measuring birefringence was used. Test results of fiber orientation are expressed by the values of birefringence ($n_e - n_o$), where higher values mean better orientation of fibers in the axial direction. Measuring of the degree of orientation was performed on a polarizing microscope – MIN8, LOMO, Russia, with five measurements for each sample. results of which are shown as a mean value.
- Fiber diameter (d) was determined on a microscope – MIN8 using an eyepiece with a measuring reticle with magnification of 5x, while the objective magnification is 40x.
- The content of end groups in polyamide textured yarn was determined using the end group titration method. The content of end groups is calculated on the basis of the volume in 1 ml of the solution used for the titration of 1 g of polyamide. The content of end groups is expressed in mmol/g of fiber. The total content of end groups is determined as the sum of amino and carboxyl groups (mmol/g of fiber).

Results and discussion

Figures 3 and ***4*** show the dependence of structural characteristics of textured yarn on the texturing parameters T (°C) and V_i (m/min), at a constant ratio of the disk surface speed to the linear yarn speed of a) D/Y = 1.9 & b) D/Y = 2.1.

A higher value of the D/Y ratio means a higher disk speed to yarn speed, and thus higher torque is transferred to the yarn, producing higher yarn twist; but also sliding between the yarn and disk may occur. Yarn tension instability is increased, producing higher variations of textured yarn characteristics [8-11]. Therefore in this work the upper limit of the D/Y ratio is carefully selected as 2.1.

According to the literature, the degree of crystallinity of textured yarn decreases with a torque increase, set by the D/Y ratio [12-14]. Increasing the texturing

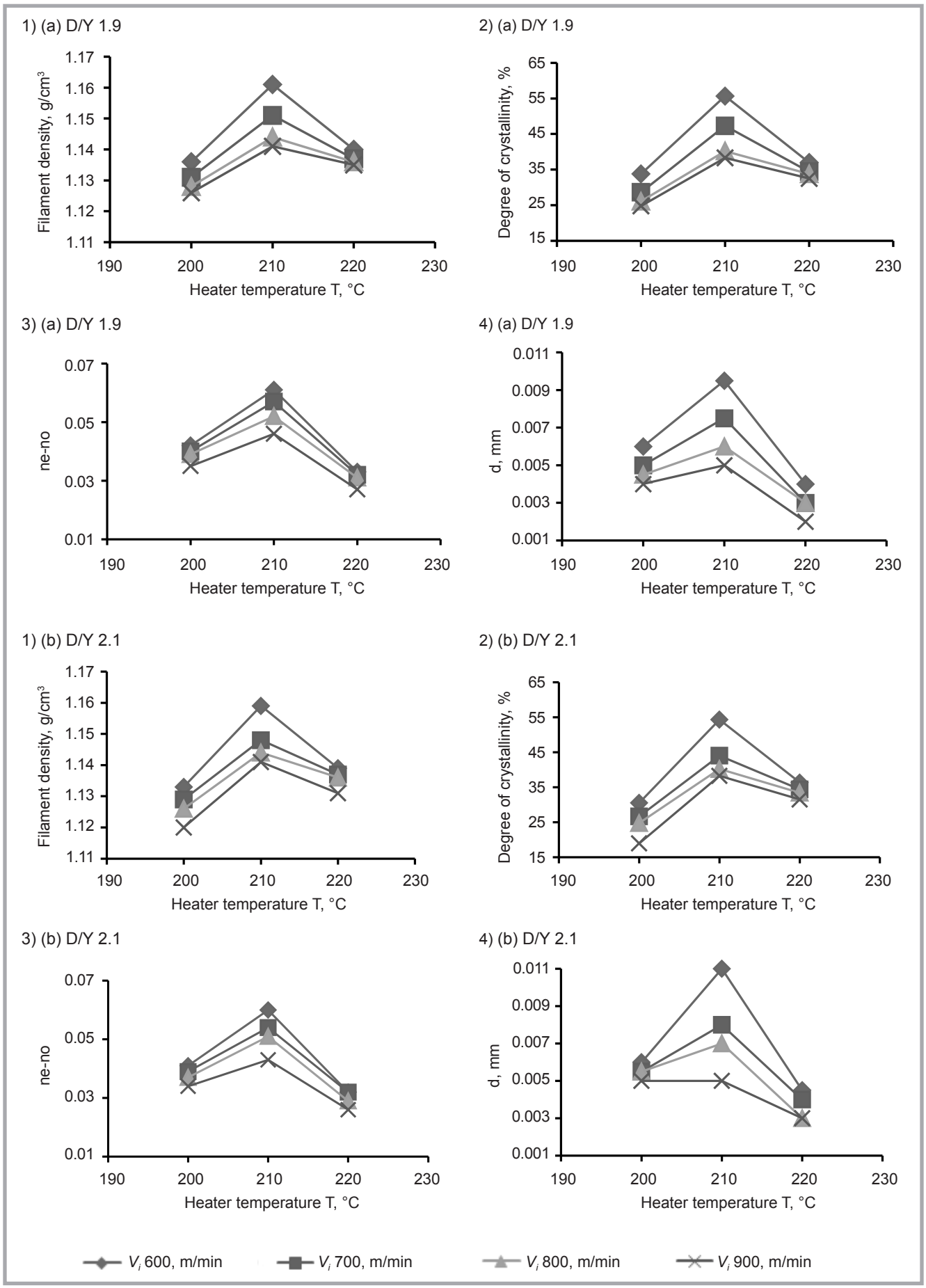

Figure 3. *Dependence of textured yarn structural characteristics on texturing conditions T, °C, V_i, m/min, D/Y: 1) filament density, g/m³, 2) degree of crystallinity, %, 3) birefringence n_e-n_o, and 4) single filament diameter d, mm.*

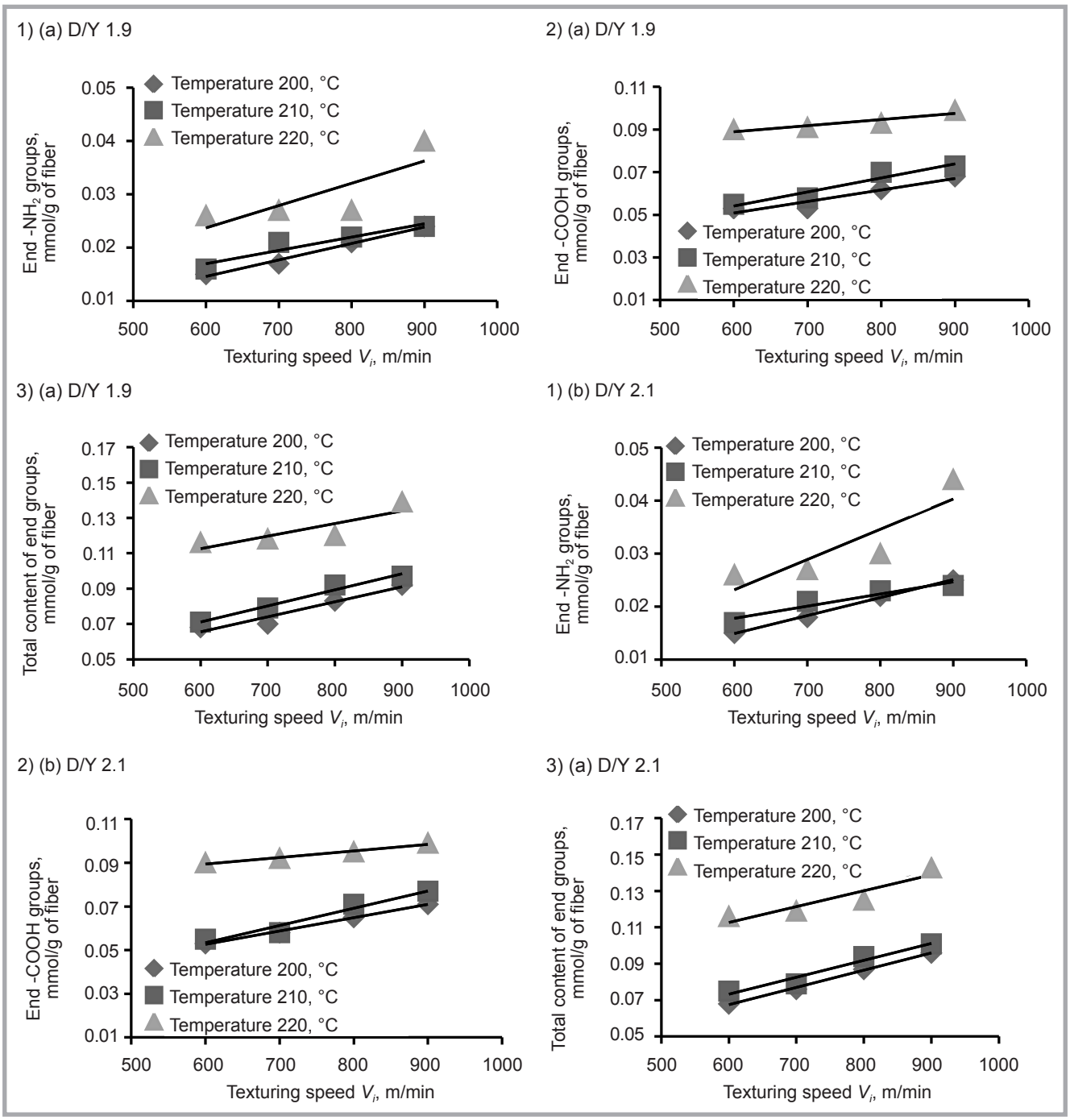

Figure 4. *Dependence of textured yarn structural characteristics on texturing conditions T, °C, V_i, m/min, D/Y: 1) end -NH_2 groups, mmol/g of fiber, 2) end -COOH groups, mmol/g of fiber, 3) total content of end groups, mmol/g of fiber.*

speed means a shorter yarn contact time in the heater and shorter yarn cooling time [15]. As the yarn contact time is increased, heat transfer from the heater to the filament increases as well as the crystallization time. Test results for the yarn degree of crystallinity at various texturing parameters range as follows:

- D/Y = 1.9, T 200 °C, variation of V_i 600-900 m/min, 33.786-24.786 (%)
- D/Y = 2.1, T 200 °C, variation of V_i 600-900 m/min, 30.571-24.786 (%)
- D/Y = 1.9, T 210 °C, variation of V_i 600-900 m/min, 55.643-38.286 (%)
- D/Y = 2.1, T 210 °C, variation of V_i 600-900 m/min, 54.357-38.286 (%)
- D/Y = 1.9, T 220 °C, variation of V_i 600-900 m/min, 37,000-32.500 (%)
- D/Y = 2.1, T 220 °C, variation of V_i 600-900 m/min, 36.357-31.536 (%)

Test results for the degree of orientation of filaments at various texturing parameters range in the following values:

- D/Y = 1.9, T 200 °C, variation of V_i 600-900 m/min, 0.042-0.035
- D/Y = 2.1, T 200 °C, variation of V_i 600-900 m/min, 0.060-0.037
- D/Y = 1.9, T 210 °C, variation of V_i 600-900 m/min, 0.06-0.046
- D/Y = 2.1, T 210 °C, variation of V_i 600-900 m/min, 0.060-0.043
- D/Y = 1.9, T 220 °C, variation of V_i 600-900 m/min, 0.033-0.027
- D/Y = 2.1, T 220 °C, variation of V_i 600-900 m/min, 0.032-0.026

Analysing the results obtained, it is obvious that increasing the D/Y ratio of the surface disk speed to the linear yarn speed at heater temperatures 200, 210 and 220 °C marginally reduces the degree of crystallinity and orientation. Also the crystallinity and orientation decrease with increasing speed V_i, as expected.

At heater temperatures T of 20-210 °C, the degree of crystallinity increases, which can be explained by the higher mobility of molecular segments and better ability to crystallise. With a further increase in the heater temperature up to 220 °C, test results show that the degree of crystallinity and orientation decline as a result of structural disorientation, increased mobility of macromolecular segments and increased content of low molecular fractions [16].

Yarn density is reduced by lowering and increasing the temperature below or above 210 °C, which is taken as the standard for the yarn fineness tested. Density values were obtained indirectly through the crystallinity degree and therefore show similar variations when texturing parameters change.

According to literature, two factors have an impact on the degree of orientation, torsion stress, assisting disorientation of molecular chains and tensile stress, which further orientates parts of molecular chains in crystal and amorphous regions. The impact of these two factors becomes more significant with temperature increase and better mobility of molecules [17-19].

At 200 °C lower values of the orientation degree were obtained (for D/Y 1.9: 0.042-0,035, D/Y 2.1 0.060-0.037), which is explained by the lower mobility of macromolecular segments at lower heater temperature. Increasing the temperature up to 220 °C results in lower orientation (for D/Y 1.9: 0.033-0.027, D/Y 2.1: 0.032-0.026) due to increased mobility of macromolecules and not enough time for structure relaxation.

It is known that in yarn texturing, variations in the cross section circular shape and diameter occur depending on the texturing method, with deviation of the POY filament diameter appearing, which can be considered as a deformation [20]. In the friction false twist method, with external and internal friction, twisting is achieved with rotating surfaces of various shapes (cylinders, disks etc). Newer models of friction mechanisms with external friction consist of friction disks set on three shafts forming an equilateral triangle (triplet system). Different profiles of disk working surfaces and disk surface curvature allow the yarn to pass over a bigger or smaller surface of the disk's upper part, being of different slope relative to the bottom part of the disk. Friction surface curvature has an important role in the texturing process [21-23].

In our case, the disk configuration is 1-4-1, and disks are ceramic with s diameter of 52 mm and thickness of 9 mm. Based on test results, increasing the D/Y ratio slightly affects the increase in diameter of individual filaments. However, by increasing the speed V_i, the diameter is marginally reduced at 200 and 220 °C, while a decrease in diameter at 210 °C is significantly higher. The resulting changes in the diameter of individual filaments can be regarded as damage caused while moving the yarn over the friction surface, in dependence on the contact geometry of the yarn and disk, the disk type, torsion. On the other hand, they can be explained through structural changes. With a higher orientation degree of individual filaments, the fiber structure is more orderly and can be more difficult to deform, which may influence the occurrence of lower variation in the diameter of the individual filaments. This is not the case with our testing because at a heater temperature of 210 °C and various speeds, we observed higher variations in diameter, which can be related to the disorientation of the yarn structure, breaking intermolecular bonds, and to the fiber being deformed more easily, resulting in higher variations in the diameter of individual filaments. Variations in diameter are higher at a D/Y ratio of 2.1 than with a D/Y ratio of 1.9.

Table 1. *Linear equations and correlation factors of dependent variables (end -NH_2 groups, end -COOH groups, total content of end groups) and the independent variable texturing speed V_i (m/min) for varying heater temperatures T, °C and constant D/Y ratio 1.9.*

Heater temperature T, °C	End -NH_2 groups, mmol/g of fiber	Linear equation	Correlation factor
200	Independent variable texturing speed V_i, m/min	$y = 3 \cdot 10^{-5} x - 0.004$	$R^2 = 0.985$
210		$y = 3 \cdot 10^{-5} x + 0.002$	$R^2 = 0.899$
220		$y = 4 \cdot 10^{-5} x - 0.001$	$R^2 = 0.658$
Heater temperature T, °C	**End -COOH groups, mmol/g of fiber**	**Linear equation**	**Correlation factor**
200	Independent variable texturing speed V_i, m/min	$y = 5 \cdot 10^{-5} x + 0.018$	$R^2 = 0.900$
210		$y = 7 \cdot 10^{-5} x + 0.014$	$R^2 = 0.930$
220		$y = 3 \cdot 10^{-5} x + 0.071$	$R^2 = 0.862$
Heater temperature T, °C	**Total content of end groups, mmol/g of fiber**	**Linear equation**	**Correlation factor**
200	Independent variable texturing speed V_i, m/min	$y = 8 \cdot 10^{-5} x + 0.014$	$R^2 = 0.938$
210		$y = 9 \cdot 10^{-5} x + 0.016$	$R^2 = 0.974$
220		$y = 7 \cdot 10^{-5} x + 0.07$	$R^2 = 0.744$

Test results show (***Figure 4***) that heater temperature has a high effect on the total content of end groups. It was observed that the end group content increases slightly with an increase in the texturing speed at heater temperatures of 200 and 210 °C. However, at 220 °C and by increasing the speed, there are much greater variations in the content of end groups, especially end -NH_2 groups. By changing the D/Y ratio from 1.9 to 2.1, the increase is slight, which can be explained as a degradation of molecular chains due to the heater temperature. In the further yarn treatment process, especially dyeing, an increased content of end groups is of great importance, being responsible for the quality of yarn dyeing, primarily level dyeing.

The results of correlation factors for end -NH_2 groups, end -COOH groups, the total content of end groups as dependant variables and the texturing speed V_i (m/min) as an independent variable, at the three texturing temperatures of 200, 210 and 220 °C and at a D/Y ratio of 1.9 are shown in ***Table 1.*** Linear equations of the corresponding dependences are given.
On the basis of the correlation factors, the best correlation of the parameters tested V_i (m/min) and T °C was found for:

- Content of end -NH_2 groups – heater temperature 200 °C ($R^2 = 0.985$)
- Content of end -COOH groups – heater temperature 210 °C ($R^2 = 0.930$)
- Total content of end groups – heater temperature 210 °C ($R^2 = 0.974$)

The results of correlation factors for end -NH_2 groups, end -COOH groups, the

Table 2. *Linear equations and correlation factors of the dependent variables (end -NH_2 groups, end -COOH groups, total content of end groups) and independent variable texturing speed V_i (m/min) for varying heater temperatures T(°C) and constant D/Y ratio 2.1.*

Heater temperature T, °C	End -NH_2 groups, mmol/g of fiber	Linear equation	Correlation factor
200	Independent variable texturing speed V_i, m/min	$y = 3 \cdot 10^{-5}x - 0.005$	$R^2 = 0.996$
210		$y = 2 \cdot 10^{-5}x + 0.004$	$R^2 = 0.920$
220		$y = 6 \cdot 10^{-5}x - 0.011$	$R^2 = 0.778$
Heater temperature T, °C	**End -COOH groups, mmol/g of fiber**	**Linear equation**	**Correlation factor**
200	Independent variable texturing speed V_i, m/min	$y = 6 \cdot 10^{-5}x + 0.016$	$R^2 = 0.996$
210		$y = 8 \cdot 10^{-5}x + 0.006$	$R^2 = 0.949$
220		$y = 3 \cdot 10^{-5}x + 0.071$	$R^2 = 0.978$
Heater temperature T, °C	**Total content of end groups, mmol/g of fiber**	**Linear equation**	**Correlation factor**
200	Independent variable texturing speed V_i, m/min	$y = 9 \cdot 10^{-5}x + 0.010$	$R^2 = 0.996$
210		$y = 9 \cdot 10^{-5}x + 0.017$	$R^2 = 0.955$
220		$y = 9 \cdot 10^{-5}x + 0.060$	$R^2 = 0.862$

total content of end groups as dependant variables and the texturing speed V_i (m/min) as an independent variable, at three texturing temperatures of 200, 210 and 220 °C and at a D/Y ratio of 2.1 are shown in ***Table 2.*** Also given are linear equations for these dependencies.

Based on the correlation factor, the best correlation between the parameters V_i (m/min) and T °C is found at:

- Content of end -NH_2 groups – heater temperature 200 °C ($R^2 = 0.996$)
- Content of end -COOH groups – heater temperature 200 °C ($R^2 = 0.996$)
- Total content of end groups – heater temperature 200 °C ($R^2 = 0.996$)

Conclusions

The variation in texturing parameters (T, V_i and D/Y) affects yarn structural changes in varying degrees. The results obtained indicate the following conclusions:

- Variations in heater temperature have a higher impact than those in the texturing speed and D/Y ratio on the degree of crystallinity, density and orientation of individual filaments, as was expected.
- Decreasing the heater temperature below 210 °C, which was taken as a standard for yarn with the fineness tested, induces a reduction in the degree of crystalinity and orientation. This can be explained by the reduced mobility of molecular segments and ability to crystallise.
- Increasing the heater temperature above 210 °C again induces a reduction in the degree of crystalinity and orientation, which can be explained by the increased mobility of molecular segments and insufficient time for relaxation of residual stresses in the yarn as a result of texturing, and by the increase in low molecular fractions.
- Variations in the diameter of individual filaments can be considered a result of damage originating in the texturing process as well as due to the disorientation of structural elements.
- The heater temperature has a more significant impact on the content of end groups than the texturing speed and D/Y ratio. Under the influence of temperature higher than 210 °C, for yarn of the fineness tested, variations in the content of end groups can be explained as a shortening of macromolecular chains due to high heater temperature.
- The correlation factor between the texturing speed and the content of end groups generally declines with a heater temperature increase.

In the texturing process, thermoplastic filament is exposed to mechanical stress at a high temperature, resulting in the changing of its structural properties. The degree of these variations is closely correlated to both the properties of the filament and to the process parameters (heater temperature, texturing speed, ratio of disk surface speed and linear yarn speed – D/Y, strain degree and disk combination in the friction assembly) [24-28].

Based on the analysis of test results, a recommendation for optimal texturing parameters can be derived which would produce the best yarn structural characteristics. For the texturing of polyamide multifilament with a fineness of 22f07x1 dtex, the optimum heater temperature is up to 210 °C, the texturing speed 750-800 m/min, the D/Y ratio 1.9; the tension in the texturing zone (strain) 1.305 and in the winding zone 0.954.

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