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SETTING OF POLYESTER FIBERS*

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INTRODUCTION. Thermal setting of synthetic fibers is mainly used for dimensional stabilization, improving some properties and removing internal stresses evolved during spinning and drawing.. The main mechanism of setting is establishment of new conformation balances of polymeric chains in fibers deformed during their formation. In semi-crystalline polymers this process is accompanied by re-crystallization leading to more per-

fect crystallites with higher melting temperatures. In the loose state the chains retraction and disorientation in amorphous regions occurs. These processes are macroscopically visible as shrinkage. The structural changes during setting are dependent on the state of fibers (pre stress) and setting conditions (humidity, temperature and time of treatment). In this work the dependence of selected polyester fibers on the setting temperature is investigated.

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PET FIBERS. The polyethylene terephthalate (PET) fibers represent polyesters having rigid benzene ring in its backbone. These fibers were patented by Whinfield and Dickson in 1941. The consumption of polyester fibers is presently about 46 % of world fibers production.

PET exhibit glass transition T_g (about 77...80°C), crystallization temperature (180...190°C) and melting point (256°C). The elastic modulus of crystalline regions of PET in direction parallel with chain axis is 108 GPa. PET fibers are well known for their high performance properties, resistance and good tensile properties. The geometrical structure of the terephthalate unit is in the Fig. 1.

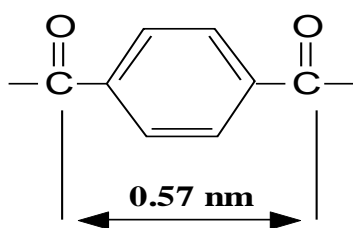


Fig. 1. Dimensions of terephthalate unit

The molar volume of PET $V_{PET} = 144 \text{ cm}^3/\text{mol}$. The amorphous density of PET $\rho_{aPET} = 1333 \text{ kg/m}^3$. The crystalline density of PET $\rho_{cPET} = 1440 \text{ kg/m}^3$. Total energy of secondary bonds is 1.37 kJ/mol. Undrawn fibers are practically amorphous. The semi-crystalline structure appears mainly during thermal setting in isometric or isotonic state. Changes of PET structure after setting are schematically shown in Fig. 2.

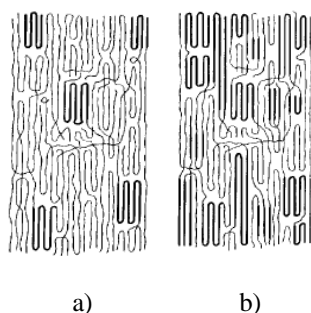


Fig. 2. Structure of PET before (a) and after (b) setting

Fibers after setting have degree of crystallinity about 0.4, orientation factor of crystalline phase is $f_c > 0.95$ and orientation factor

of amorphous phase is $f_a = 0.6$. Basic structural units are relatively strong micro fibrils having diameter 10...15 nm and length 103 nm. These units are assembled into fibrils having diameter 30...45 nm.

Very important role is played by oriented non crystalline phase (TTM) – „taut tie molecules“. Portion of TTM is around 0.1...0.05. This phase is responsible for mechanical properties of PET fibers.

EXPERIMENTAL PART. The staple unmodified dull polyester fibers with fineness 3.3 dtex were used. Dry thermal setting in oven (loose state) at three temperatures 140, 170 and 200 °C during 15 min. was realized. The changes of mechanical thermal and surface characteristics were investigated. The thermal behavior was measured on the DSC 6 (Perkin Elmer) under nitrogen atmosphere at rate of heating 20 K/min. Ultimate mechanical properties were measured on the dynamometer Vibrodyn 400 (Lenzing Instruments). Dynamical mechanical measurements were realized on the apparatus DMA DX04T (RMI Ltd.). Fiber surfaces were investigated by using electron raster microscope VEGA TS 5130 (Tescan) having resolution 3.5 nm. Fiber images have resolution 512 x 512 pixels.

RESULTS AND DISCUSSION. The typical DSC thermogram of polyester fibers after setting is shown in the Fig. 3.

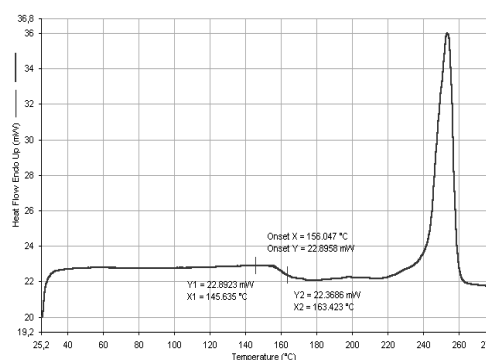


Fig. 3. Thermogram of polyester fiber (DSC)

Typical bend in the exothermic direction is indicator of setting temperature. Location of this bend is called effective setting temperature or start of recrystallization (T_{rc}). The location of maxima on melting peak corresponds to the melting temperature (T_m). Both these characteristics are given in the Table 1.

It is visible that values T_{rc} are about 15K higher than selected setting temperature. The setting temperature of original fibers (setting during fiber production) was about 134°C. The melting temperature is practically independent on the setting temperature. The temperatures corresponding to the maximum of loss tangents $T_{tan \delta}$ measured at frequency 0.1 Hz on the DMA apparatus are presented in the Table 1. The evaluation of $T_{tan \delta}$ is shown in the Fig. 4.

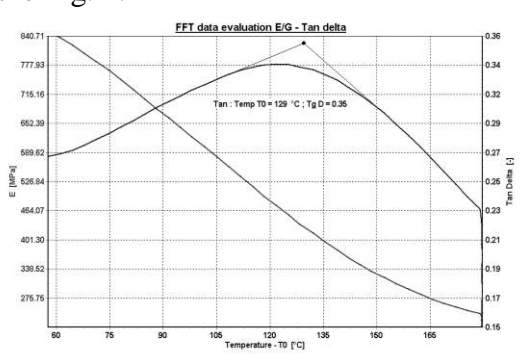


Fig. 4. Evaluation of $T_{tan \delta}$ from dynamic mechanical measurements (DMA)

The increase of setting temperature leads to the decrease of $T_{tan \delta}$ due to the increase of mobility in the amorphous regions. Mean values of tenacity and break elongation are given in the Table 1 as well. The tenacity slightly decreases and deformation at break increases with setting temperature increasing. The distribution of tenacity can be approximated by the two parameter Weibull distribution. Parameters of the Weibull distributions were practically unchanged by setting temperature. The statistical nature of fibers break is therefore not dependent on the setting temperature. The deformation energy to break is characterized by factor $SF = \sigma\sqrt{\epsilon}$, where σ is tenacity and ϵ is fiber breaking elongation. It is visible that SF factor increases with the increasing of setting temperature.

Table 1. Thermal and mechanical characteristics of fibers after setting

Setting temperature	T_{rc} [°C]	T_m [°C]	$T_{tan \delta}$ [°C]	Tenacity [mN]	Break elongation [%]	Factor SF
Original sample	148,8	255,7	112	153,9	34,5	903
140°C	156,3	254,5	110	153,9	35,7	917
170°C	185,1	255,4	90	152,7	37,7	937
200°C	215,8	255,4	81	145,9	42,3	948

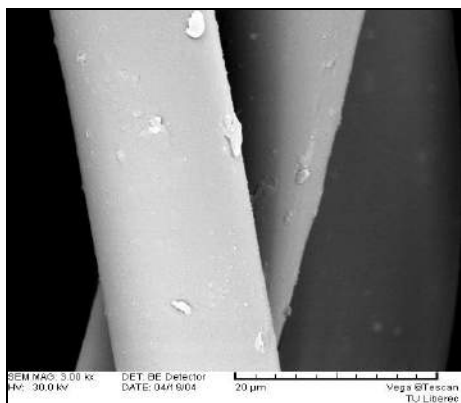


Fig. 5. Original fiber

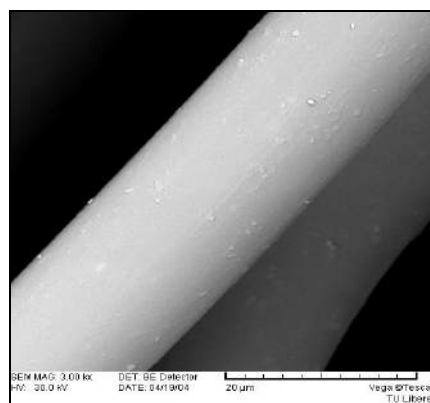


Fig. 6. Cleaned fiber

The surface of fibers was characterized by electron scanning microscopy. Surface of original fiber is shown in the Fig. 5. The oligomers on the surface of original fibers were firstly removed by repeated extraction in boiled ethanol at temperature 78.3°C.

Surface of cleaned fibers is shown in the Fig. 6. These cleaned fibers were then treated at above mentioned setting conditions. The effective setting temperatures T_{rc} from DSC thermograms were unaffected by setting temperature. Surfaces of cleaned fibers after set-

ting are presented in the Figs. 7, 8 and 9. It is clear that oligomers creation is equilibrium reaction. After their removing from surface

and setting the oligomers are reappear on the surface due to diffusion.

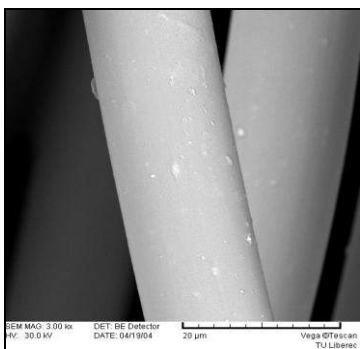


Fig. 7. Setting at 140°C

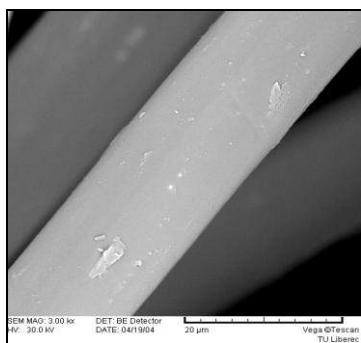


Fig. 8. Setting at 170°C

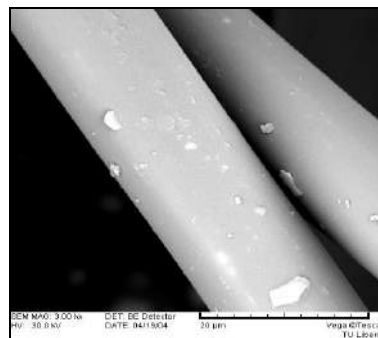


Fig. 9. Setting at 200°C

The breaking zone of fibers before and after setting is shown in the Figs. 10 up to 13. Increase of setting temperature leads to the

increase of plastic flow portion at fiber break and corresponds to the increase of breaking elongation or deformation work to break.

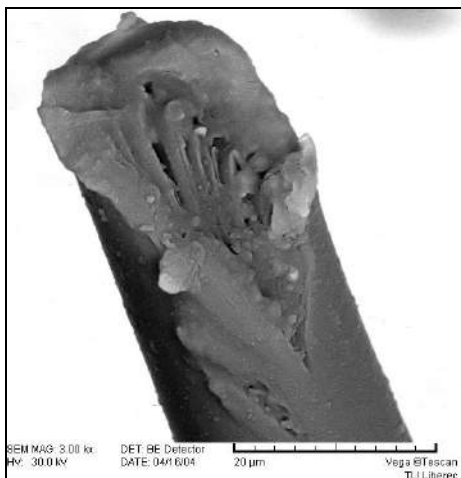


Fig. 10. Breaking zone of original fiber

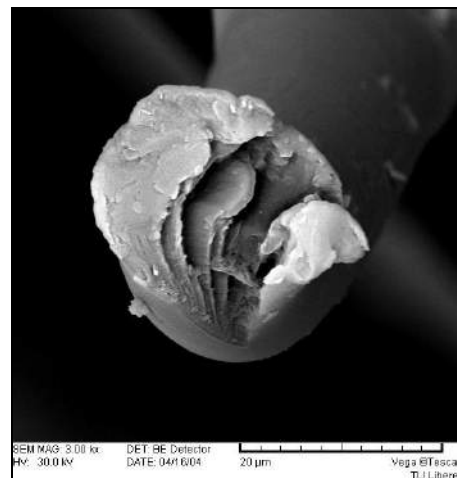


Fig. 11. Breaking zone (setting at 140°C)

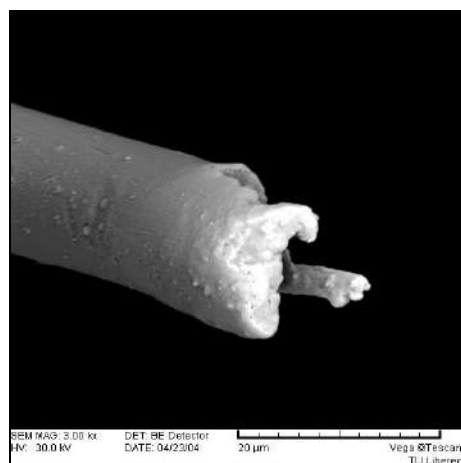


Fig. 12. Breaking zone (setting at 170°C)

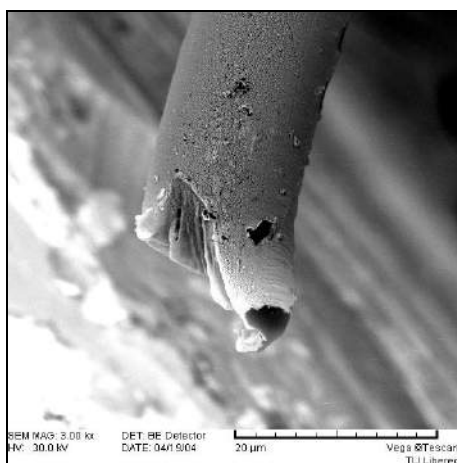


Fig. 13. Breaking zone (setting at 200°C)

CONCLUSION

The influence of setting temperature on the behavior of polyester fibers was investigated. Results of measurements are in accordance with assumption that increase of setting temperature leads to the increase of amount and quality of crystalline phase and its separation from amorphous phase. Direct consequence of these structural changes is increase

of chains mobility in amorphous phase and increase of plastic deformation portion at fiber deformation. The oligomers are appeared on the surface of fibers after setting.

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