

Thermomechanical and Shape Memory Performances of Thermo-sensitive Polyurethane Fibers

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(Received February 9, 2017; Revised November 29, 2017; Accepted December 4, 2017)

Abstract: This study is the first step to investigate usability of shape memory polyurethane (SMPU) fibers for smart garment applications. SMPU fibers were spun by wet spinning process and chemical/mechanical characterization was carried out. SMPU solutions were prepared with two different concentrations (20 % and 25 %) and three different coagulation bath concentrations (0 %, 1 % and 3 %) were used for determining optimum spinning parameters. For investigating influences of spinning process on crystal structure, mechanical, thermal and shape memory performances of fibers, X-ray diffraction (XRD), differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA), scanning electron microscopy (SEM) and mechanical tests were conducted. DSC and DMA analysis results show that shape memory polyurethane fibers have a glass transition temperature about 35-40 °C which is suitable for body temperature. Moreover, SMPU fibers showed good tensile performance with an average tenacity of 1.38 cN/dtex and elongation at break of 350 %. Thermo mechanical test results showed that, all shape memory fibers have good shape memory effect with recovery and fixity ratios up to 91 % and 71 % respectively.

Keywords: Shape memory polyurethane, Wet spinning, Characterization, Glass transition temperature, Shape memory behavior

Introduction

Shape memory polymers (SMPs), which are a group of smart materials, have been taking more attention for different end uses because of their performance in returning their original (permanent) shapes from a temporary shape under external stimulus such as heat, light, pH, moisture [1-5]. When compared with other shape memory materials, SMPs have remarkable properties such as lightweight, good processing ability, high shape recoverability and large range of shape recovery temperature. As a result of the mentioned advantages, they have been widely studied and used in academic researches and industrial applications especially in last two decades [6-8]. Nowadays, thermal induced SMPs use in different application fields such as textile, other engineering areas and biomedical devices [9-13].

In terms of chemical structure, SMPs are phase-segregated linear multiblock copolymers composed of two segments, a hard segment and a soft segment, which are responsible for a frozen phase and a reversible phase. The reversible phase transformation of the soft segment is responsible for the shape memory effect and serves as a molecular switch. The glass transition temperature (T_g) or the crystalline melting temperature (T_m) of soft segment is used as the shape transition temperature (T_{trans}) of SMPs [14-16]. Mechanical properties of SMPs are strongly affected by their micro-morphology [17-19]. The shape memory properties depend on many factors such as chemical structure, composition and molecular weight of SMPs, distribution of hard and soft

segments [13,15]. Several polymer systems such as polyurethane, *trans*-polyisoprene (TPI), poly(styrene-co-butadiene), polynorbornene have been reported to possess shape memory properties [6].

Shape memory polyurethanes (SMPUs) have the advantage of broad switch temperature (glass transition or melting temperature) by changing soft and hard segment types and contents [3,20]. SMPUs are phase separated polymers due to the thermodynamic incompatibility between hard and soft segments [1] and this characteristic enable remarkable shape memory effect. Soft segment content is the decisive parameter affecting phase separation process, and consequently shape memory behavior of SMPU. Numerous works have been reported on synthesis parameters and resulting shape memory performance of segmented SMPUs. Most of these studies focused on changing the types or molecular weight of the soft segment during polymer synthesis to change glass transition temperature of polymer [14,21-23].

Because of good processability and tailorable mechanical properties (elasticity, strength and abrasion resistance), among many other application areas, SMPUs are suitable for textile processes such as fiber spinning and finishing to fabricate smart textiles with self-adaptability to body and environmental temperature [1,24-26]. Therefore, various applications of SMPU in different forms such as fiber, yarn, film and foam have been studied in cases of production and characterization. Especially, in some studies, various shape memory fibers were produced in order to enhance mechanical properties and they were compared with commercial elastane fibers such as lycra, XLA and nylon. Results of these studies show that shape memory fibers have a lower breaking

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strength and acceptable strain performances compared with the conventional synthetic fibers. In recent years, many researchers have focused on development of functional fibers and smart textile products with shape memory effects such as core spun yarn, breathable textiles, wound dressing materials, artificial tendons and protective garments [1-4,7,9,10,25,27,28]. Some of these studies have related to fibers spun by wet, melt or electro spinning techniques with shape memory polyurethane, and investigations about their mechanical, thermal and shape memory performances [1,4,6,14,20,24,28]. Shape memory polyurethane (SMPU) is the most commonly used among SMPs for a wide range of applications with different forms in smart applications such as thermal protective clothing, sportswear, underwear, gloves, socks and aesthetic applications [15].

In this study, a spinning optimization was carried out by producing six types of SMPU fibers under different conditions (solution and coagulation bath concentrations) by wet spinning process. Performances of the spun fibers having switching temperatures suitable for human body were investigated. Moreover, influences of solution and coagulation bath concentrations on chemical, mechanical and shape memory performances of fibers were analyzed.

Experimental

Materials

Pellet-type MM-3520 SMPUs ($M_n=24473$; $M_w=116483$) were purchased from SMP Technologies Inc. (Japan) for production of shape memory fibers (SMF). As informed by the manufacturer, the polymer was composed of amorphous soft segments and it has a nominal T_g of 35 °C which is used as T_{trans} temperature ($T_{trans}=T_g$). It consists of, diphenylmethane-4,4'-diisocyanate, adipic acid, ethylene glycol, ethylene oxide, polypropylene oxide, 1,4-butanediol and bisphenol A. Shape memory polymer solution was prepared with N, N-dimethylformamide (DMF) (Sigma-Aldrich) according to a proceeding study [1] as solvent.

Production of SMPU Fibers

Shape memory fibers were spun by wet spinning method with DMF as solvent. After some pre-trials, solid concentrations of SMPU solution in DMF were adjusted to 20 % and 25 % to meet the viscosity requirements for wet spinning process. SMPU solution was extruded through single spinneret capillary hole horizontally through a coagulation bath with arranged concentrations (0 %, 1 % and 3 % DMF) to diffuse out the solvent with a spinning speed of 3.2 m/min. Mono-filament which was formed in coagulation bath was taken up to a second bath including water for removal of residual DMF. Then, SMPU filament was wound with 90 rpm on a cylindrical bobbin. Totally, six type of SMPU fibers with different parameters were

Table 1. Production details and mechanical characteristics of SMPU filaments

| Yarn code | Polymer concentration in solution (%) | Coagulation bath concentration (%) | Linear density (dtex) |
|-----------|---------------------------------------|------------------------------------|-----------------------|
| SMPU200 | 20 | 0 | 167 |
| SMPU201 | 20 | 1 | 111 |
| SMPU203 | 20 | 3 | 250 |
| SMPU250 | 25 | 0 | 84 |
| SMPU251 | 25 | 1 | 83 |
| SMPU253 | 25 | 3 | 139 |

produced (Table 1). The internal stress can be stored in crosslinking structure of fibers during the wet spinning process. In order to release the internal stress caused by the velocity difference among the rollers in the drying and winding process, heat treatment at 50 °C were applied on the produced shape memory fibers.

Characterization of Thermal Properties

Thermal properties of the SMPU polymer and fibers were determined using a differential scanning calorimeter (DSC) device with nitrogen as purge gas. First, SMPU sample was cooled to -30 °C at the cooling rate 10 °C/min, then the polymer was heated from -30 °C to 240 °C at 10 °C/min heating rate. Pellet type SMPU polymer was also investigated by DSC. The heat flow change with increasing temperature was recorded [29].

Dynamic Mechanical Analysis

The dynamic mechanical properties of the shape memory fibers were determined by using dynamic mechanical analysis (DMA) device at a frequency of 2 Hz. The gauge length for each fiber between the clamps was 15 mm and temperature was scanned from -120 °C to 200 °C with 2 °C/min heating rate with liquid nitrogen as a coolant gas [29]. Beside the information about shape memory fibers including storage modulus, loss modulus and phase angle $\tan(\delta)$, glass transition temperature of fibers were also obtained by dynamic mechanical analyses.

Characterization of Crystal Structure

X-ray diffraction (XRD) analysis is a useful tool for obtaining information about crystalline structure of semi-crystalline polymers with a range of inter-atomic distances from 0.1 to 5 nm [15]. The X-ray diffractometer (Bruker D8 Advance, Germany) was used to characterize the crystal structure of shape memory polyurethane fibers. Experiments were performed at 40 V, 40 mA current with a radiation wavelength of 1.542 Å. Spectra were obtained in θ -2 θ step scan mode with a scan range of 10-60 ° with scanning step size was 0.02 ° and step time was 1 second.

Morphology of Shape Memory Fibers

The surface morphology and cross section of shape memory fibers were characterized by scanning electron microscopy (SEM, Fei Quanta FEG 250, USA). A single fiber of the produced shape memory fiber was attached onto the SEM sample holder. The accelerated voltage of the electron beam was 5 kV and images of fibers were recorded.

Mechanical Property Test

Tensile tests of the SMPU fibers was carried out with a Lloyd Tensile Testing Machine (LR5K) according to ASTM D2256. The gauge length was 25 mm and the tensile speed was 25 mm/min. All tests were conducted under standard atmospheric conditions (20±2 °C and 65±2 RH%).

Shape Memory Property Test

The thermo-mechanical cyclic tensile test was conducted to investigate the shape memory behavior of shape memory fibers according to a preceding study [20]. Test was performed on Lloyd LR5K tensile tester (Lloyd Instruments, UK) within a temperature controlled chamber. The fiber gauge length was 25.4 mm. Schematic representation of a typical stress-strain behavior during the thermo-mechanical cycling test is shown in Figure 1. Steps of the typical thermo-mechanical cycle test procedure for shape memory fibers are as follows: I) The fiber was initially stretched to 100 % elongation at 40 °C with a speed of 100 mm/min, which is above the glass transition temperature (switch temperature) of fiber. II) The sample was cooled down to 22 °C and temperature was maintained for 15 minutes to fix temporary elongation. III) The upper clamp was returned to original position and the fiber shrank from 100 % strain (ϵ_m) to strain after unloading at 22 °C (ϵ_u) because of instant elastic recovery. IV) Sample was heated to 40 °C again to allow shape memory recovery enabling the fiber elongation returning to residual strain (ϵ_p).

After the cycle was completed, another cycle began and a

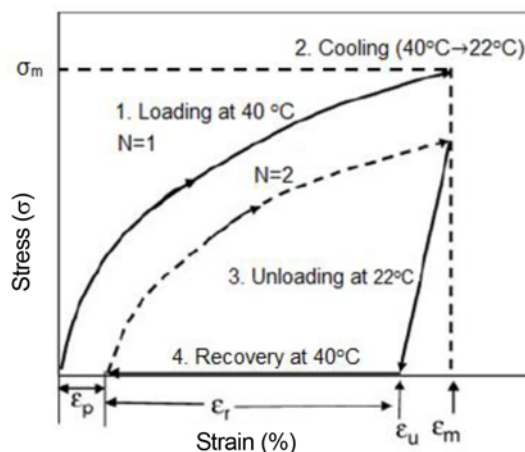


Figure 1. Thermomechanical cyclic tensile test path.

total of four cycles (N) were carried out in order to investigate the shape memory effect of fibers. The shape fixity ratio (R_f) and the shape recovery ratio (R_r) which are used to characterize the shape memory effect of a material were defined in the reported literature [7,20]. The shape fixity ratio (R_f) and shape recovery ratio (R_r) were calculated using the following equations;

$$\% \text{ Shape fixity } (R_f) = \frac{\epsilon_u(N)}{\epsilon_m} \times 100$$

$$\% \text{ Shape recovery } (R_r) = \frac{\epsilon_m - \epsilon_p(N)}{\epsilon_m - \epsilon_p(N-1)} \times 100$$

All the mechanical and shape memory performance test results were analyzed by parametric statistical analysis methods to put forward conclusions about performances of shape memory fibers and relationships between fibers which were spun with different conditions. SPSS 21.0 Statistics Software (SPSS Inc., USA) was used for statistical analysis of the results.

Results and Discussion

Thermal Property Analysis of Shape Memory Fibers

The glass transition temperature of the shape memory polyurethane and spun fibers were determined by using differential scanning calorimetry (DSC) and results are shown in Table 2. Generally, glass transition temperature is determined from the second heating cycle to provide T_g value independent of the thermal history during processing. In the second heating scan, glass transition of soft segment of shape memory polyurethane fibers was determined between the range 36.43-37.37 °C. In addition, glass transition temperature (T_g) of shape memory polyurethane in pellet form was determined as 37.69 °C which is very close to the value provided by manufacturer (35 °C). This glass transition is used as the switch temperature to fix the shape of the fiber (below and above the thermal transition temperature) [29]. Results of SMPU fibers enable end uses

Table 2. DSC analyses results for shape memory samples [29]

| Sample code | 1st Heating glass transition temp. (T_g) (°C) | Cooling glass transition temp. (T_g) (°C) | 2nd Heating glass transition temp. (T_g) (°C) |
|-------------|---|---|---|
| SMPU200 | 29.26 | 35.26 | 37.37 |
| SMPU201 | 22.10 | 33.45 | 37.03 |
| SMPU203 | 20.97 | 34.09 | 36.43 |
| SMPU250 | 20.80 | 35.26 | 36.58 |
| SMPU251 | 22.24 | 34.70 | 36.95 |
| SMPU253 | 21.79 | 32.33 | 36.81 |
| SMPU pellet | 26.17 | 33.96 | 37.69 |

related to clothing or body related products such as smart fabrics enhancing thermal comfort.

Dynamic Mechanical Analysis of Shape Memory Fibers

The storage modulus, loss modulus and phase angle results of shape memory fibers obtained from DMA analysis are shown in Figure 2, Figure 3 and Figure 4, respectively. Elasticity of shape memory fibers is directly related to density of crosslinks contained in the structure and elasticity property is reduced with increasing crosslink density [30]. According to storage modulus curves, increasing difference in storage modulus between plateau regions obtained on before and after T_g value means a reduction in the amount of crosslinking. In this case, storage modulus of SMPU251 and SMPU250 which were spun with 25 % polymer concentration in solution and 1 % and 0 % concentration of coagulation bath respectively, have sharp decreases at 27 °C and highest storage modulus were obtained for these fibers. As shown in Figure 2, SMPU251 and SMPU250 have the lowest crosslink density, accordingly, breaking elongation values were the highest for SMPU251 during tenacity test [29].

Loss modulus is related to the hysterisial energy dissipation and it shows mobility of polymer chains. High and wide loss modulus relate with mechanical properties of material such as impact resistance. It means that, material have better mechanical properties with increasing width of loss modulus curve [30]. According to loss modulus curves of shape memory fibers, SMPU250 and SMPU251 which were spun with 25 % polymer concentration in solution, 0 % and 1 % concentration in coagulation bath respectively, have better loss modulus, hence higher breaking tenacity values. Moreover, SMPU201 which was spun with 20 % polymer concentration in solution and 1 % concentration in coagulation bath has the lowest loss modulus confirming its breaking tenacity values.

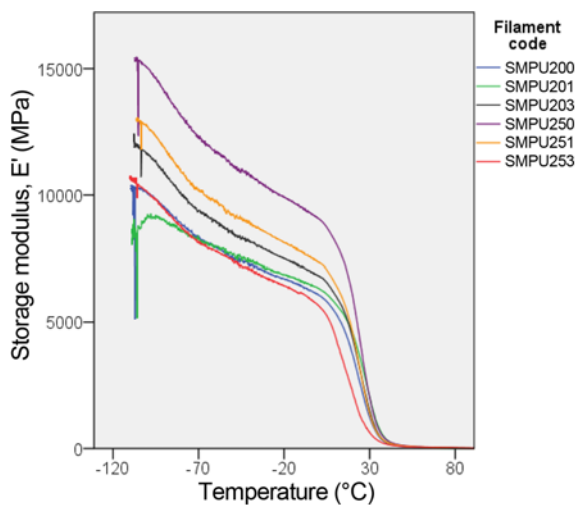


Figure 2. Storage modulus of shape memory fibers detected by DMA [29].

Besides, the loss modulus peak of fibers locating at 38-40°C is due to glass transition of soft segment phase. The loss modulus has a sharp decrease at higher temperature than T_g . This means that, fibers can be easily deformed at temperatures above T_g and shape can be fixed at temperature below T_g [29].

Figure 4 shows that $\tan\delta$ peak which is associated with the glass transition temperature of shape memory fibers range between 35.7-40.5 °C. It is generally accepted that the transition temperature measured by DMA is slightly higher than that obtained from DSC as the working principles of DSC and DMA are different [24]. Confirming literature [24,31], there is a difference of 2-3 °C between glass transition temperatures measured by DSC and DMA. The peak height of $\tan\delta$ provides information about the mobility of molecule

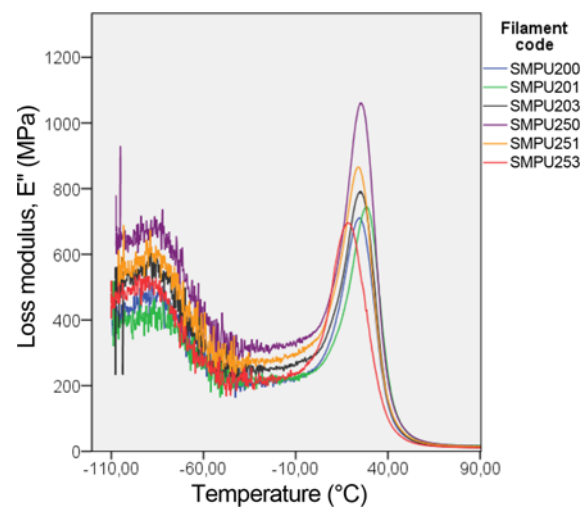


Figure 3. Effect of temperature on loss modulus of shape memory fibers [29].

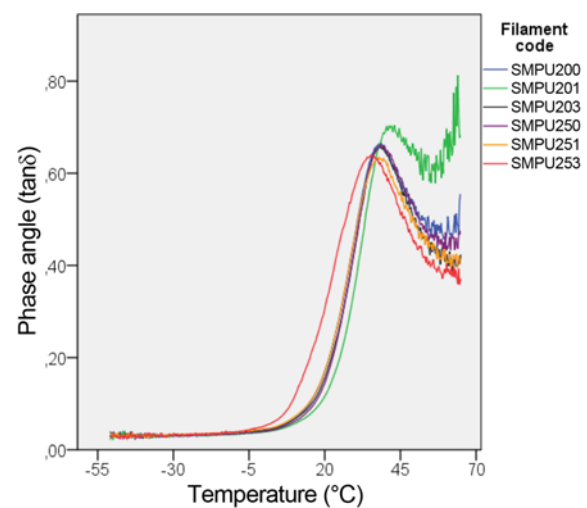


Figure 4. Phase angle curves of shape memory fibers detected by DMA [29].

chains of the materials and $\tan\delta$ peak width give information about homogeneity of crosslinking. The mobility of molecular chains in the material structure rises with increasing peak height of $\tan\delta$. The width of $\tan\delta$ gives idea about heterogeneity of material crosslinking. When phase angle curves ($\tan\delta$) of shape memory fibers obtained by DMA analysis are observed (Figure 4), SMPU201 fiber which is spun with 20 % polymer concentration in solution and 1 % concentration of coagulation bath has the highest peak value. Consequently, its chain mobility is more than the other fibers. Moreover, SMPU251 fiber which is spun with 25 % polymer concentration in solution and 1 % concentration of coagulation bath has the lowest chain mobility. Besides, SMPU251 and SMPU253 fibers which are spun with 25 % polymer concentration in solution, 1 % and 3 % concentration of coagulation bath, respectively, have more heterogeneous structures than the others [29].

Crystallinity of Shape Memory Fibers

The crystalline structures of shape memory fibers were studied by XRD. The results of XRD are shown in Figure 5 and results of parameters were summarized in Table 3. The XRD profiles of fibers demonstrated a broad peak at $2\theta=21-23^\circ$. All fibers showed broad scattering between $10-30^\circ$. The presence of small crystallite size which is scattered in

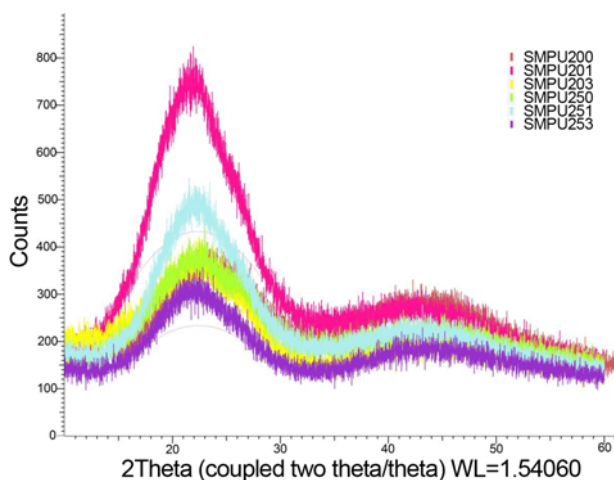


Figure 5. XRD diffraction patterns of SMPUs.

Table 3. The XRD results of peak separations of SMPUs

| Fiber code | Peak position, 2θ ($^\circ$) |
|------------|---------------------------------------|
| SMPU200 | 23.263 |
| SMPU201 | 22.003 |
| SMPU203 | 22.436 |
| SMPU250 | 23.043 |
| SMPU251 | 21.546 |
| SMPU253 | 22.810 |

structure, amorphous region or relatively short-range ordered of hard segments may have caused this results. Therefore, the degree of crystallinity of shape memory fibers was not calculated. However, high shape memory effect obtained from thermomechanical cycling test shows that the fibers have sufficient hard segment ratio. Similarly, there are some studies which reported low crystallinity and unclear peak on XRD scatter of SMPU filaments [14,28].

Morphology of Shape Memory Fibers

The surface morphology and cross-section of SMPU fibers were studied by a Fei Quanta FEG 250 SEM. SEM images of SMPU fibers were recorded at magnifications of 2000x and the surface and cross-section images are shown in Figure 6. When the surface morphology are examined, grooves can be seen on the surface of fibers which formed during spinning process. SMPU200 and SMPU250 which were spun with 20 % and 25 % polymer concentration in solution and 0 % concentration of coagulation bath, respectively, have slightly smoother surface than other fibers. Generally, SMPU fibers have similar non-circular cross-section except SMPU250 and SMPU251 which were spun with 25 % polymer concentration in solution and 0 % and 1 % concentration of coagulation bath (they have a corrugated cross-section).

Mechanical Properties of Shape Memory Fibers

The tensile test results of shape memory fibers are summarized in Table 4. Shape memory polyurethane fibers showed remarkable tensile properties with breaking tenacity between 0.62-2.21 cN/dtex and breaking elongation between 319-374 % when compared with other studies which spun fibers by wet spinning having an average tenacity 1 cN/dtex and 80-380 % strain. Although this tenacity values are low than synthetic fibers such as polyester, elastane, it is sufficient for further fabrication processes such as knitting. The Young's modulus of shape memory fibers at different temperature are also summarized in Table 4. It can be seen that Young's modulus of fibers were decreased with increasing temperature.

According to the statistical analysis results of tensile test, polymer content in solution had a positive effect on the tenacity values of fibers. As shown in Figure 7, fibers which were produced with high polymer concentration (25 %) show better tenacity performance than other fibers which were spun with 20 % concentration. SMPU250 and SMPU251 which were spun with 25 % polymer concentration in solution and, 0 % and 1 % concentration of coagulation bath, respectively, had the highest tenacity values while SMPU200 and SMPU203 had lowest values. This result is consistent with the results obtained from loss modulus curves of DMA analyses. Results of tensile test showed that strain properties of shape memory fibers, although not statistically significant ($p>0.05$), were influenced by polymer

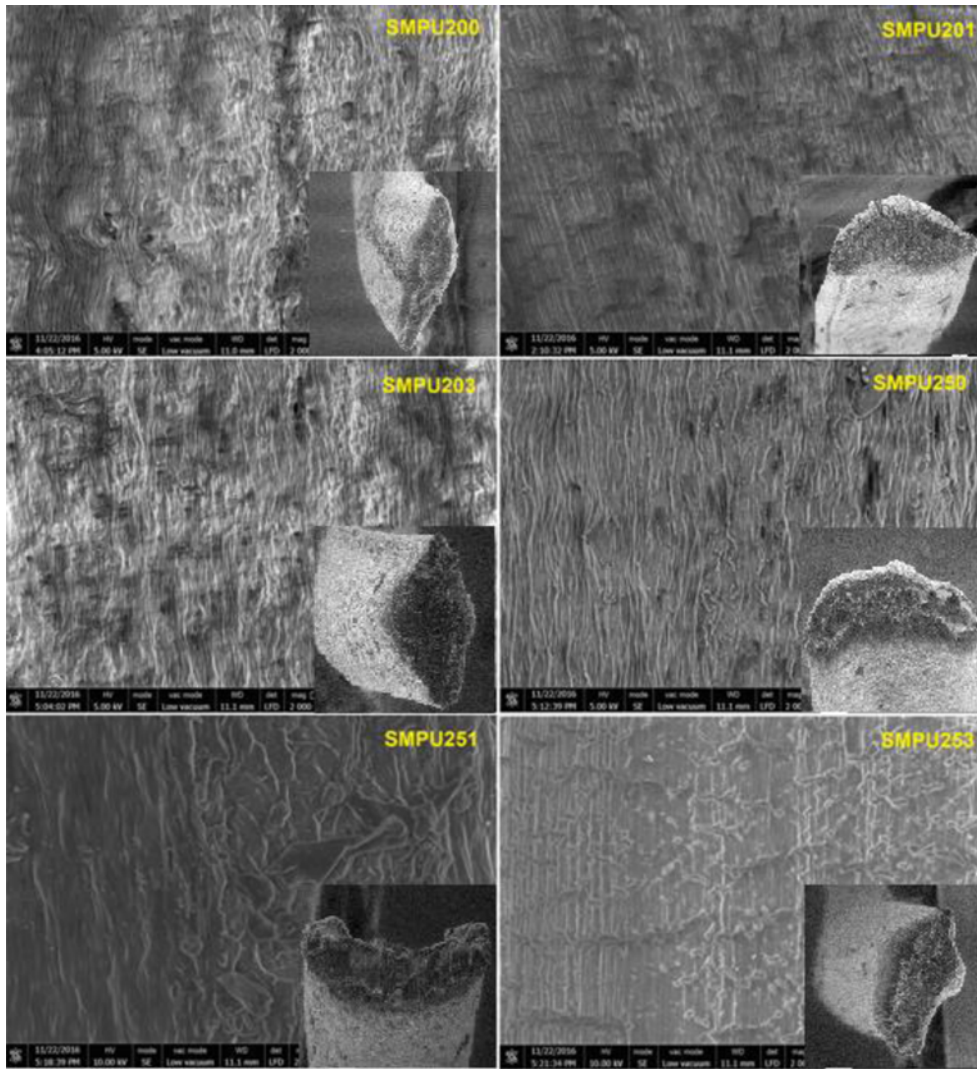


Figure 6. Surface and cross-sectional images of shape memory fibers.

Table 4. Tensile properties of shape memory fibers

| Filament code | Linear density (dtex) | Strain (%) | Tenacity (cN/dtex) | Young's modulus (GPa) | |
|---------------|-----------------------|------------|--------------------|-----------------------|-------|
| | | | | 20 °C | 40 °C |
| SMPU200 | 167 | 362.2 | 0.76 | 3.72 | 0.22 |
| SMPU201 | 111 | 373.9 | 0.90 | 4.56 | 0.33 |
| SMPU203 | 250 | 322.9 | 0.62 | 4.35 | 0.24 |
| SMPU250 | 84 | 319.2 | 2.18 | 5.96 | 0.33 |
| SMPU251 | 83 | 361.2 | 2.21 | 4.56 | 0.24 |
| SMPU253 | 139 | 360.6 | 1.39 | 2.21 | 0.17 |

concentration of spinning solution. SMPU201 fiber which was spun produced with 1 % solvent concentration in coagulation bath and 20 % polymer concentration, showed the highest strain performance among other shape memory

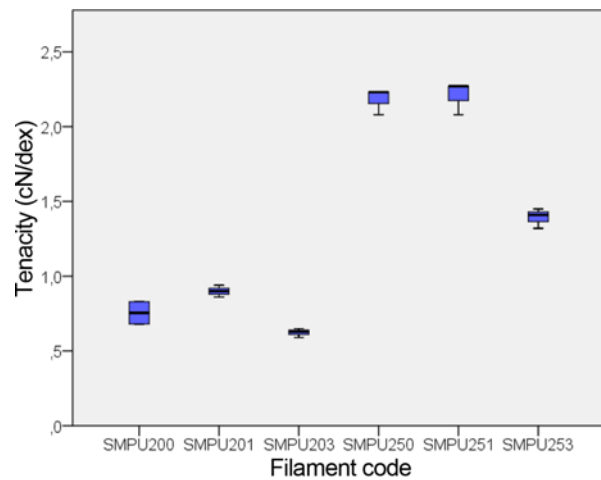


Figure 7. Boxplot diagram of tenacity values of shape memory fibers.

fibers. It was found that the fibers which were produced in this study, having average tenacity at 1.38 cN/dtex, had higher tenacity values than fibers produced in similar studies by wet or melt spinning with shape memory polyurethane. As a general evaluation, it can be said that fibers produced in similar studies [7,28,32] having 80-380 % strain, SMPU fibers spun in this study exhibit significant strain with an average of 350 % and good tenacity performances. These high tensile values are important for production of fabrics which require sufficient strength and also for production method such as knitting or weaving.

Shape Memory Performance of Fibers

The stress-strain curves of shape memory fibers obtained with thermo-mechanical cycling test are shown in Figure 8 for evaluating the differences of stress-strain behavior of fibers between cycles. Generally, shape memory fibers show similar behaviors in all cycles. However, as clearly seen in Figure 8, SMPU200 and SMPU201 which were spun with

20 % polymer concentration in solution, 0 % and 1 % concentration of coagulation bath respectively have the lowest stress values at 100 % strain. In addition, the stress values of fibers at 100 % strain were decreased in subsequent cycles, as expected. When compared with stress values of 1st cycle, there was an average decrease in stress values by 33.8 % in the 2nd cycle, 39.4 % in the 3th cycle and 41.4 % in the 4th cycle. Despite decreasing stress values, the shape recovery ratio of fibers increased after 1st cycle as a result of orientation of soft and hard segments as given in Table 5. This can be explained by orientation of soft and hard segment of shape memory fibers under heat and stress treatment [14].

Results of thermo-mechanical cyclic tensile test are summarized in Table 5. Shape memory fibers have average fixity ratio of 72.5 % and recovery ratio of 91.5 % in the first cycle. The fibers show up to 95 % recovery ratio and 71 % fixity ratio in the other two cycles. This results show that, spun fibers have good shape memory effect confirming

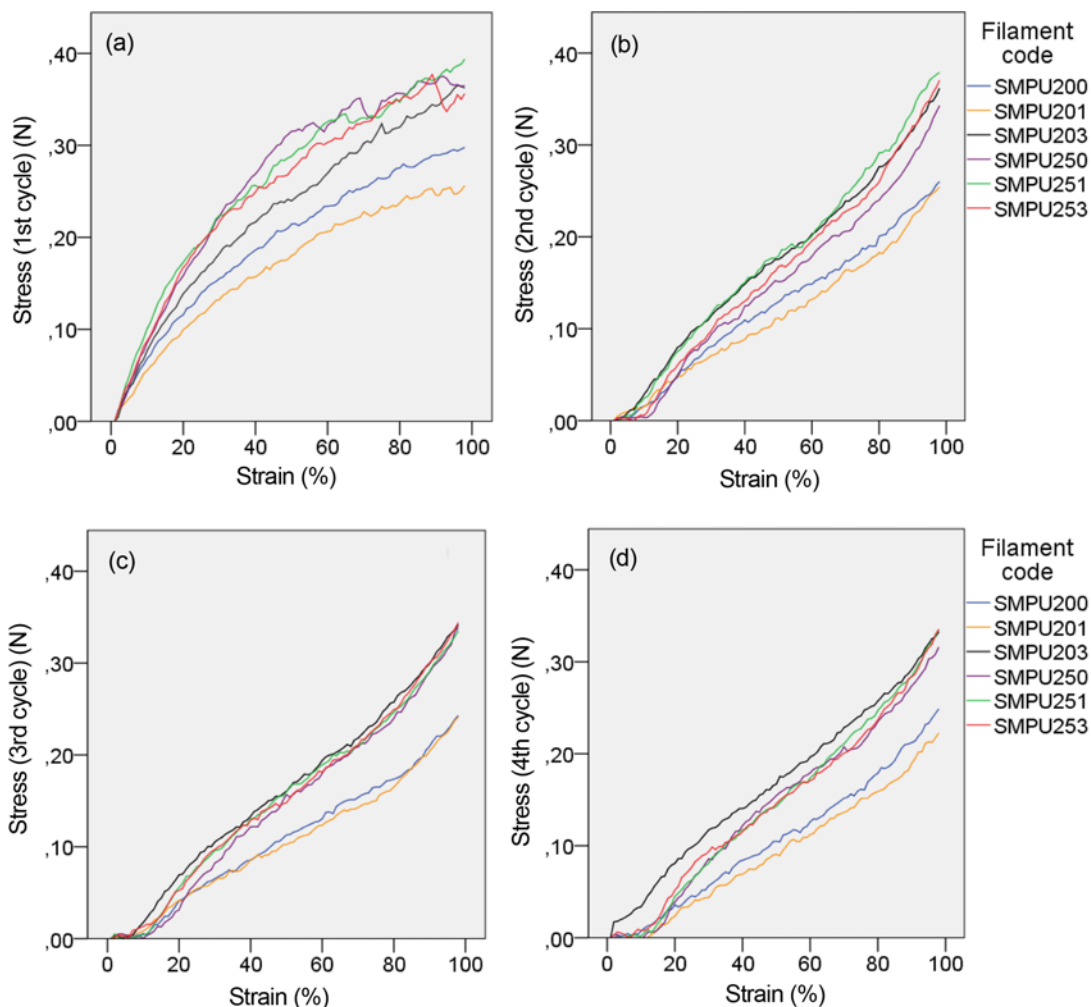


Figure 8. Stress-strain curves of shape memory fibers obtained from thermo-mechanical cycling test.

Table 5. Thermomechanical properties of shape memory fibers under drawing temperature [29]

| Cycle code | Shape recovery ratio (%) | Shape fixity ratio (%) |
|------------|--------------------------|------------------------|
| 1 | 91.5 | 72.5 |
| 2 | 95.2 | 71.3 |
| 3 | 95.9 | 71.6 |
| 4 | 97.3 | 71.3 |

preceding studies [6,20]. Besides, it is also believed that shape memory effect of fibers sufficient for developing smart textiles that change their shape at body temperature. Because of changes in molecular orientation and crystallization, there were differences between the first and corresponding cycles. The shape memory effect and stress-strain behavior of fibers were very similar except for the first cycle confirming preceding studies [20,29]. The differences between first and other cycles are thought to be due to reorganization of fiber molecules involving molecule orientation, crystallization, or a weak point broken during elongation. The shape memory fibers cannot be completely fixed to their temporary shapes due to the instantaneous elastic recovery during removal of the external stress, consistent with the results obtained in a similar thesis study [1] which were evaluated shape memory performance of shape memory polyurethane fibers. At the same time, the fibers were not completely recovered to their original shapes due to molecular shear and weak breaks that occurred during elongation [1].

The shape fixity ratio and shape recovery ratio results obtained from thermomechanical cyclic tensile tests of shape memory fibers are shown in Table 6. According to average of thermomechanical test results, all shape memory fibers showed similar shape memory performances. However, SMPU251 fiber which is spun with 25 % polymer concentration in solution and 1 % concentration of coagulation bath has

slightly higher recovery and fixity ratio values, demonstrating a better shape memory performance than other fibers.

Conclusion

Shape memory polyurethane (SMPU) fibers were successfully produced with wet spinning method with different solvent and coagulation bath concentrations. It can be concluded that glass transition temperature (T_g) of soft segment parts, at which the shape change could be triggered, were found between the range 36-40 °C according to DSC and DMA analyses and solvent concentrations in coagulation bath did not have a remarkable effect on glass transition temperature of fibers. Produced fibers may be used for clothing or products related to human body such as sportswear, wound dressing, surgical protective garments, waterproof and breathable fabrics, socks, wrinkle-free fabrics. Shape memory polyurethane fibers showed good tensile performances, especially the fibers spun with high polymer solution concentration and without solvent in coagulation bath. Shape memory fibers have remarkable shape memory performances that they have a fixity ratio about 72.5 % and recovery ratio up to 91.5 % in the first cycle. The fibers show up to 95 % recovery ratio and 71 % fixity ratio in the other corresponding cycles. These values are quite sufficient for shape memory effect necessary especially for end uses including a deformation and body temperature change. Shape memory polymers have great potential for developing functional and smart textile products such as breathable, waterproof, modified structures.

Acknowledgements

This study was funded by a project of Suleyman Demirel University Scientific Research Projects Coordination Unit (4393-D2-15). We want to thank for their support.

Table 6. Shape recovery and shape fixity ratios of shape memory fibers

| Fiber code | Shape memory effect | 1st cycle (SD) | 2nd cycle (SD) | 3rd cycle (SD) | 4th cycle (SD) |
|------------|--------------------------|----------------|----------------|----------------|----------------|
| SMPU200 | Shape recovery ratio (%) | 91.0 (2.94) | 93.6 (2.23) | 93.0 (6.44) | 97.6 (0.30) |
| | Shape fixity ratio (%) | 73.1 (3.84) | 71.5 (3.09) | 73.7 (4.84) | 72.1 (4.41) |
| SMPU201 | Shape recovery ratio (%) | 91.0 (2.94) | 95.1 (1.26) | 96.3 (1.24) | 96.2 (1.32) |
| | Shape fixity ratio (%) | 73.1 (5.00) | 71.5 (3.09) | 73.7 (2.54) | 72.1 (2.54) |
| SMPU203 | Shape recovery ratio (%) | 91.7 (2.22) | 95.8 (0.10) | 97.1 (1.17) | 97.7 (0.03) |
| | Shape fixity ratio (%) | 68.6 (4.00) | 68.3 (3.47) | 67.6 (1.47) | 70.5 (2.22) |
| SMPU250 | Shape recovery ratio (%) | 90.4 (1.92) | 94.3 (1.32) | 96.3 (1.23) | 96.9 (1.25) |
| | Shape fixity ratio (%) | 71.8 (2.22) | 71.8 (2.94) | 71.5 (2.94) | 70.2 (2.88) |
| SMPU251 | Shape recovery ratio (%) | 93.0 (1.11) | 95.9 (0.05) | 97.1 (1.27) | 97.8 (0.05) |
| | Shape fixity ratio (%) | 74.4 (4.41) | 71.5 (2.78) | 70.8 (2.42) | 71.2 (3.33) |
| SMPU253 | Shape recovery ratio (%) | 91.7 (1.11) | 96.5 (1.23) | 95.7 (0.09) | 97.7 (0.05) |
| | Shape fixity ratio (%) | 72.5 (1.11) | 73.4 (1.47) | 72.1 (1.67) | 71.8 (1.11) |

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