Materials Today Communications 19 (2019) 98–105

Contents lists available at ScienceDirect

Materials Today Communications

journal homepage: www.elsevier.com/locate/mtcomm

Continuous drawing of poly(ethylene terephthalate) in ethanol and agile functionalization through infusion

Dongwoo Go[a], Wataru Takarada[a], Arun Aneja[b], Takeshi Kikutani[a,*]

[a] Department of Materials Science and Engineering, School of Materials and Chemical Technology, Tokyo Institute of Technology, 2-12-1 S8-32, O-okayama, Meguro, Tokyo, Japan

[b] Department of Engineering, East Carolina University, Greenville, NC, 27858, USA

ARTICLE INFO

Keywords:
Poly(ethylene terephthalate)
Continuous drawing
Infusion

ABSTRACT

To explore the possibility of applying organic-solvent drawing technique to accomplish a process of industrial significance, continuous drawing of as-spun PET fibers in ethanol was performed. The formation of multiple necks, infusion of ethanol into filament along with the uptake of dissolved dye was observed during the continuous drawing of PET filaments. The propagation speed of individual neck increased as the draw ratio increased, while effect of revolution speed of roller on the neck propagation speed was minimal because of the increase of the number density of neck along with the increase in processing speed. Through the monitoring of the drawing tension, it was found that the fluctuation of drawing tension is a source of instability of the drawing process. The installation of a drawing pin was discovered to be useful for the stabilization of the drawing process while increasing the amount of infused ethanol and dye uptake.

1. Introduction

Poly(ethylene terephthalate) (PET) is one of the most widely used polymer materials. PET provides wide versatility covering the range of commodity plastics to engineering plastics. Even in markets where the demand for high-performance plastics is increasing, PET is being used in many applications for its low price compared with other engineering plastics. For manufacturing PET fibers, it is necessary to have a higher-order structure of high crystallinity and high degree of orientation [1]. In order to have such a structure, drawing and annealing of melt-spun low oriented amorphous fibers either through an off-line process or an in-line spin-drawing process, or a direct high-speed melt spinning method is mainly utilized [2–5].

Inducing crystallization by using an organic solvent has been reported [6–10] as one of the methods for crystallization of PET at room temperature. It is known that the PET can be plasticized due to the effect of the organic solvent and the crystallization occurs at room temperature by lowering the glass transition temperature [11–14].

On the other hand, it has been reported that in the tensile drawing of amorphous low oriented PET fibers in an organic solvent [15–19], multiple-necking phenomenon occurs because of the reduction of surface free energy from polymer – air interface to polymer – solvent interface [15]. This drawing method is called multiple-neck drawing. In addition, infusion of an organic solvent, i.e. sucking in of organic solvent with the aid of tensile drawing, was demonstrated. The infusion phenomena occurs when a fiber is drawn in an organic solvent [16–19] without requiring long time intervals or high temperature which are typically involved in conventional diffusion methods. Crystallization of molecules by organic solvents infused into fibers has also been reported [16–19].

In our previous studies [17–19], we have performed batch-type drawing of PET filaments on mini-tensile machine using ethanol as an organic solvent. We chose ethanol, which is less toxic than other organic solvents, considering the environmental safety. It has been confirmed that in the batch-type drawing process using ethanol or ethanol/water solution, multiple-necking phenomenon occurred, yield stress and drawing stress decreased, and the natural draw ratio increased. It was also confirmed that, along with the development of molecular orientation, the solvent induced crystallization of the PET molecules occurred due to the influence of the ethanol infusion. This is a cold drawing process, and the crystallization occurs at room temperature. It should be noted that neither infusion of ethanol into PET fiber nor solvent induced crystallization occurs if PET fiber is only immersed in ethanol with no axial stress. This means that the utilization of the infusion process is more advantageous if ethanol is used instead of other stronger organic solvents.

Various applications of infusion phenomenon occurring in the organic solvent drawing process are expected [16–22]. Typical

* Corresponding author.
E-mail address: kikutani.t.aa@m.titech.ac.jp (T. Kikutani).

https://doi.org/10.1016/j.mtcomm.2019.01.001
Received 26 December 2018; Accepted 2 January 2019
Available online 03 January 2019

applications include low temperature dyeing of fiber and transport of dissolved material (imbibition) to yield desired functional properties [16–19,23,24]. Through the infusion of solvent and imbibition of dissolved dye, solvent induced crystallization occur in a single step drawing process. Other advantage of this process is that it does not require any additional process such as annealing or washing after dyeing making it an eco-friendly and sustainable strategy. A variety of functional modifications are also available to create an agile process with no down time for reactor clean-up. It is expected that it will be possible to modify or give new properties to PET fibers by using not only dyes but also drugs or modifiers as solutes.

The drawing process using the organic solvents reported so far have been conducted mainly in batch-type studies. There have been few studies on the organic solvent drawing methods for the continuous process. To explore the possibility of applying this unique behavior for an efficient industrial process, continuous drawing of PET fibers in ethanol was performed in this current research and drawing behavior was investigated in detail. Our particular interests were neck-drawing and infusion behavior in the continuous drawing process, i.e. whether the multiple necking occurs or not, whether the stable neck-drawing can be achieved, whether the enough amount of ethanol can be infused into the fiber, and how the structure development of fibers proceeds along with the infusion of ethanol. In this paper, we will concentrate on the in-situ observation of neck-drawing behavior, and the structure development behavior will be discussed in the forthcoming paper.

2. Experimental

2.1. Materials

As spun amorphous poly(ethylene terephthalate) (PET) filament was used in this study. Before fiber spinning, PET pellets were dried in a vacuum oven at 110 °C for over 12 h. The PET filament was prepared by extruding PET polymer at 300 °C from a spinneret with a single circular hole of 1.0 mm diameter. The filament was quenched at room temperature without applying cross flow air. The throughput rate was maintained at 5 g/min, and filament was wound-up on a bobbin using a winder at a velocity of 400 m/min. The diameter of as spun amorphous PET filament prepared for this study was about 105 μm, and tensile strength and elongation at break of that filament were 100 MPa and 840%, respectively. The natural draw ratio (NDR) of that filament evaluated through an ordinary tensile testing was about 4.3. Ethanol was used as the organic solvent in the solvent drawing process. In some experiments, ethanol with 0.5 wt% of Sumikaron Red SE-RPD disperse dye was used for studying the uptake behavior of solute in the solvent.

2.2. Continuous drawing process

Continuous drawing of the PET filament was carried out in ethanol using a set of rollers operating at different rotation speeds. The circumference of rollers were 317 mm. An ethanol flowing-in glass channel of the length of 300 mm was placed between the two rollers. The schematic illustration of continuous drawing with the ethanol channel is shown in Fig. 1(a). The machine draw ratio (MDR) and drawing speed were determined by adjusting the rotation speeds of a feed roller and a take-up roller. Continuous drawing of as spun amorphous filaments was carried out at various feed roller rotation speeds of 1–10 rpm and draw ratios (DR) of 2–4. In some experiments, a snubber pin was placed across the drawing line in the ethanol channel as a method to stabilize the drawing process by suppressing the tension fluctuation and inducing homogeneous neck formation. The schematic illustration of continuous drawing system equipped with a pin in an ethanol bath is shown in Fig. 1(b).

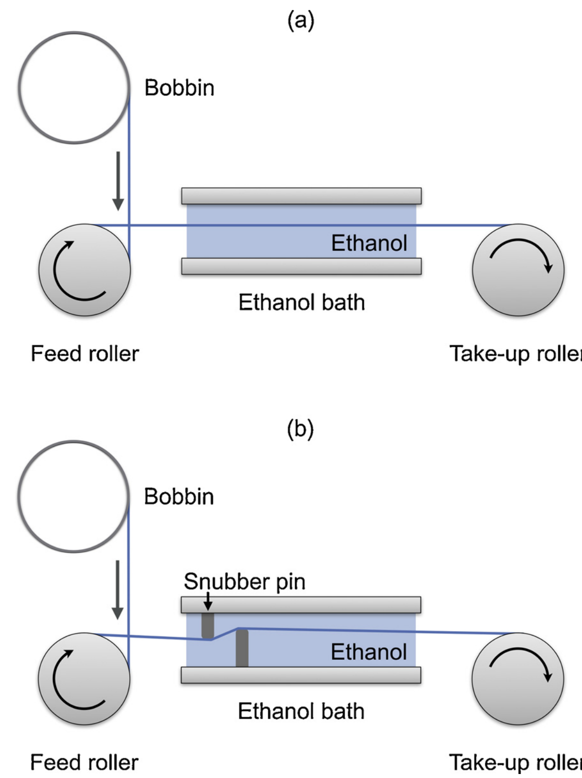

Fig. 1. Schematics of (a) continuous cold drawing system in ethanol channel and (b) drawing system equipped with a pin.

2.3. Observation and measurement

In order to evaluate the drawing stress for each drawing condition, a three-point tension meter (TTM-201, Toray Engineering Co., Ltd., Japan) was provided on the drawing line. The tension variation during the drawing process was also measured to analyse the stability of the continuous drawing process.

Observation of drawing behavior of PET filament in ethanol channel was carried out using a high-speed camera (Phantom V9.0, Vision Research Inc., USA). The locations observed by the high-speed camera were 0, 33, 66, 100, and 200 mm from the entrance of the ethanol channel. For each drawing condition, multiple-necking behavior and velocity profile were analyzed.

2.4. Evaluation of infusion

In order to confirm the infusion behavior during the continuous solvent drawing process, in some experiments, drawing was performed using 0.5 wt% dye-ethanol solution instead of neat ethanol. After drawing, the dye concentration in the drawn PET filament was analyzed utilizing a laser Raman spectrometer (NRS-5100, Jasco Inc., Japan). A laser source with the wavelength of 784.82 nm was used to minimize the effect of fluorescence from the dye molecules. The laser power was kept at about 13 ˜ 15 mW. The laser spot with the diameter of 2 ˜ 3 μm was focused on the surfaces of the drawn and undrawn part of the fiber under microscope to analyse these two parts separately.

3. Results and discussion

3.1. Tension measurement

In order to evaluate the stability of continuous drawing process, the draw line tension was measured during the drawing process without a snubber pin as shown in Fig. 1 (a). Fig. 2(a) shows the result of

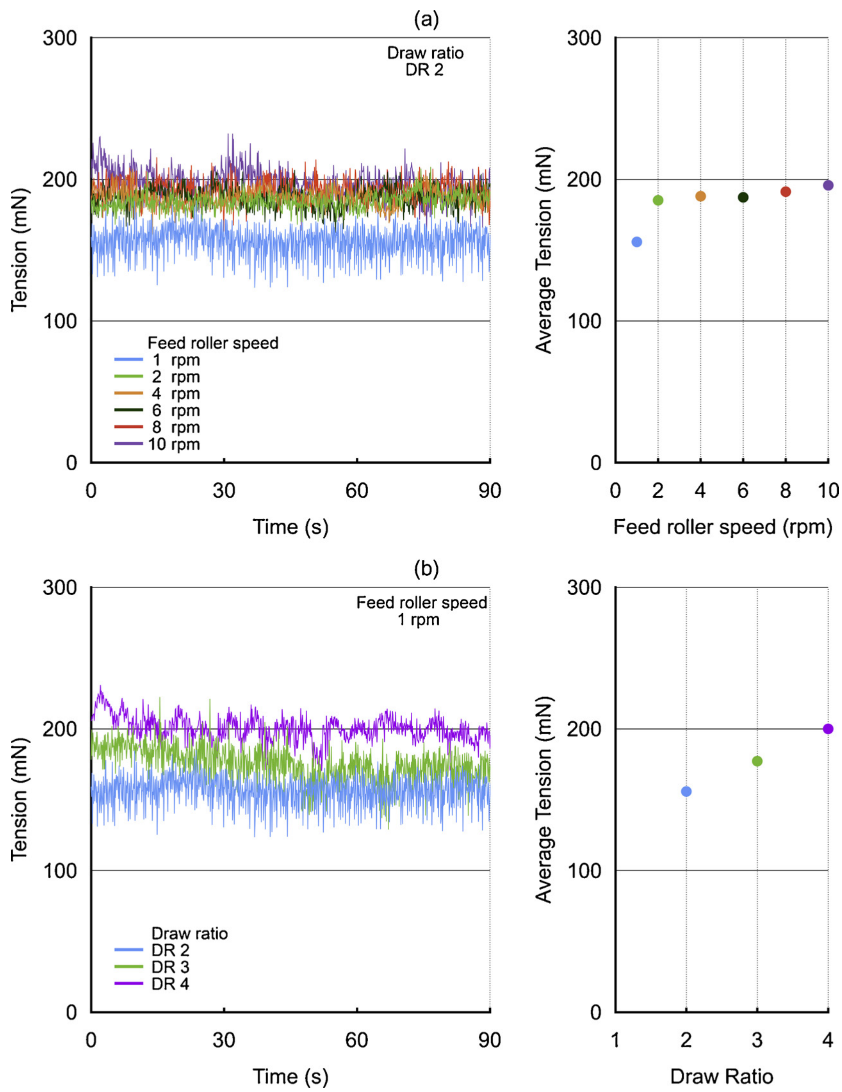

Fig. 2. Variation of drawing line tension with time for (a) different feed roller speeds and (b) draw ratios. Draw ratio and feed roller speed for Fig. 1 (a) and (b) were DR 2 and 1 rpm, respectively. These measurement was conducted without the snubber pin. Variations of averaged tension with feed roller speed and draw ratio are also shown. Colors of the tension variation data correspond to the color of symbols for the averaged values.

measuring the drawing line tension at DR 2 and at various feed roller rotation speeds. Fig. 2(b) shows the result of draw ratio versus tension for each draw ratio at the feed roller rotation speed of 1 rpm.

Significant fluctuation of drawing tension was observed for all drawing conditions. It was confirmed that the draw tension applied to the drawing line increased as the drawing speed increased. It was also confirmed that the draw tension increased as the draw ratio increased. It should be noted that in this experiment, drawing tension of 100 mN corresponds to the drawing stress of 11.5 MPa. In case of the drawing of PET filament in the air at room temperature, and at the feed roller speed and draw ratio of 1 rpm and DR 5.4, drawing stress was around 37 MPa. It can be said that the drawing stress was significantly suppressed by the use of ethanol as a drawing media.

3.2. Captured images of the drawing line

During the drawing process, the PET filament in ethanol channel was observed with a high-speed camera. The captured images are shown in Fig. 3, in which variations of drawing behavior depending on the feed roller speed and the distance from the entrance of ethanol channel are shown. Formation of multiple necks in the continuous drawing of PET filament in ethanol as in the case of batch-type drawing was confirmed from these images. The neck points were not evenly distributed along the drawing line. In other words, there was an unevenness in the starting point of multiple necks. Even with such uneven distribution of neck points, it was clearly confirmed that at a fixed distance from the entrance of ethanol channel of 33 mm, the number density of neck, i.e. number of necks per unit length, increased with the increase of feed roller rotation speed as can be seen in Fig. 3(a). It was also found that with the increase of distance from the entrance of ethanol channel under a certain drawing condition, the amount of drawn part increased, the amount of undrawn part decreased, and the number density of neck decreased as shown in Fig. 3(b).

3.3. Increase tendency of instantaneous draw ratio

During the continuous drawing with the formation of multiple necks, increase of averaged instantaneous draw ratio in a particular region of the drawing-line corresponds to the increase and decrease of the total lengths of drawn and undrawn parts, respectively, in that region. The instantaneous draw ratio, which can be defined as the ratio of the cross-sectional area of the original filament to that of the averaged cross-sectional area in a certain narrow region of the drawing line where multiple necks may exist, was obtained analyzing the local

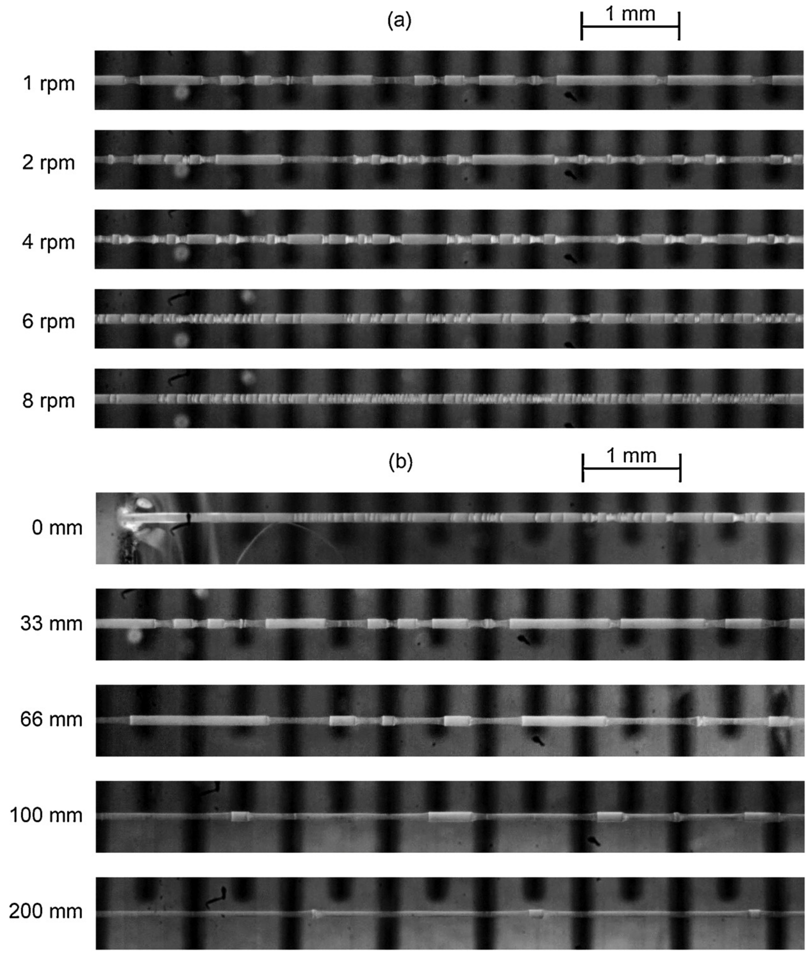

Fig. 3. High speed video images of PET filament under the drawing process in ethanol without a snubber pin. (a) : Effect of feed roller speed at a position near the entrance of ethanol channel. Distance from the entrance of ethanol channel at the left-end of the photographs is 33 mm, and draw ratio is 4. (b) : Effect of distance from the entrance of ethanol channel. Feed roller rotation speed is 1 rpm, and draw ratio is 4. The distance values shown in the figure correspond to the left-end in the individual photographs.

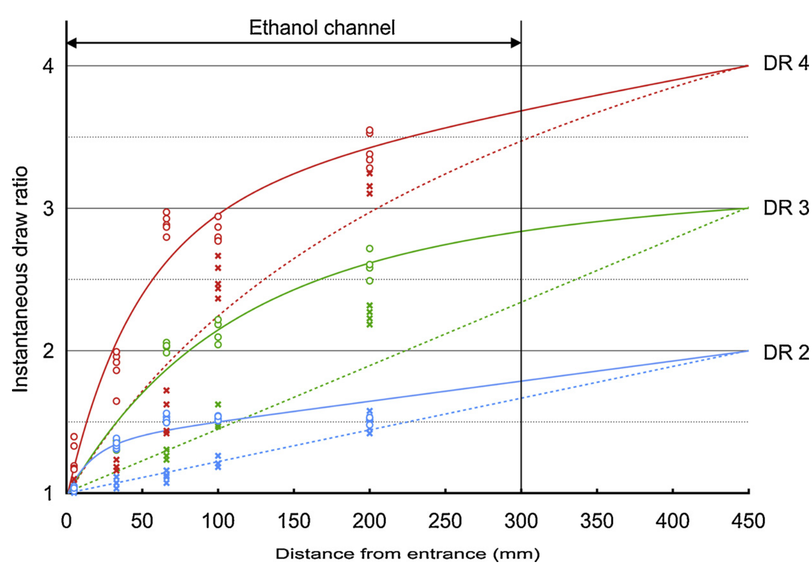

Fig. 4. Variation of instantaneous draw ratio with the increase of distance from the entrance of ethanol channel without a snubber pin. ○ symbols with full lines and × symbols with dashed lines are for the feed roller speed of 1 and 10 rpm, respectively.

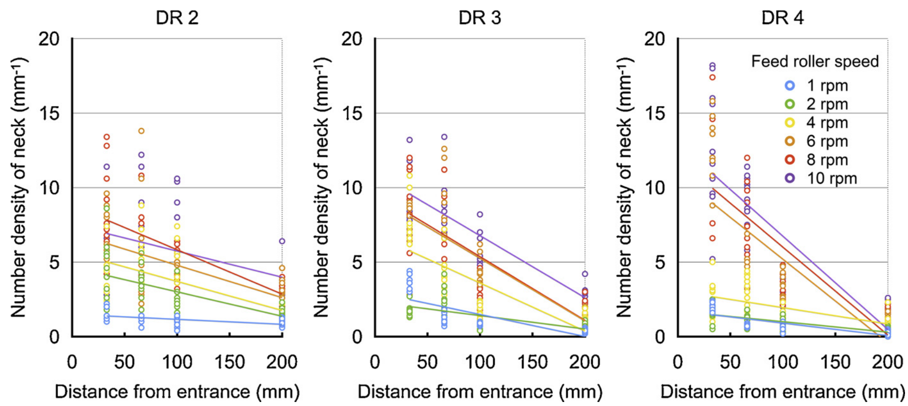

Fig. 5. Variation of number density of neck with the increase of distance from the entrance of ethanol channel without a snubber pin. The trend lines were obtained by the least square method for different feed roller speeds. Results for the draw ratios of 2, 3 and 4 are shown.

velocity of the filament in that particular region from the video image. Based on the concept of mass conservation, instantaneous draw ratio was calculated as the ratio of the local velocity to the velocity of the feed roller surface. With the increase of local filament velocity instantaneous draw ratio also increases. Fig. 4 shows the results of instantaneous draw ratio variation along the drawing line for the feed roller rotation speeds of 1 and 10 rpm, and the machine draw ratios of 2, 3 and 4. The instantaneous draw ratio, which was expected to increase to the machine draw ratio before reaching the take-up roller, gradually increased with the propagation of the necks. It was found that the instantaneous draw ratio increased more rapidly at an early stage in the ethanol bath for the slower feed roller speed. It should be noted that considering the difference in drawing speed, the local velocity gradient, i.e. elongational strain rate, was obviously higher for the feed roller speed of 10 rpm. On the other hand, at a certain distance from the entrance of ethanol channel, instantaneous draw ratio increased almost proportionally with the increase of the machine draw ratio for both feed roller speeds.

3.4. Number density of necks

Measurements of the number of necks was carried out for each observed position and each drawing condition. As a result of repeated measurements of several times under the same conditions, the number of necks per measurement showed wide distribution. However, based on trends from Fig. 5, it can be seen that the number density of neck increased rapidly when the filament entered the ethanol channel, and then decreased gradually with the increase of distance from the entrance. This was mainly because of the increase of instantaneous draw ratio, while there can be a further reduction of the number density of neck because a pair of necks at each end of the undrawn part could merge and vanish with the reduction of the length of the undrawn part. It can be seen clearly that the number density of neck increased with the increase of feed roller speed, while there was a slight increase of the number density of neck with the increase of machine draw ratio.

3.5. Neck propagation speed

In our previous paper on the batch-type drawing of PET filament in ethanol, it was revealed that the amount of ethanol infused into the filament has close correlation with the propagation speed of individual neck [18]. Considering this, the propagation speed of the neck in the continuous drawing process was estimated for each drawing condition and each position in ethanol channel.

Assuming that the lengths of undrawn and drawn parts of the filament at distance x from the entrance of ethanol channel, L_1 and L_2, change to the lengths $L_1+\Delta L_1$ and $L_2+\Delta L_2$ at distance $x+\Delta x$, and the time for the movement of filament from x to $x+\Delta x$ is Δt, then the local strain rate dV/dx can be expressed as follows:

$$\frac{dV}{dx} = \frac{\frac{(L_1+\Delta L_1+L_2+\Delta L_2)-(L_1+L_2)}{L_1+L_2}}{\Delta t} = \frac{\frac{\Delta L_1+\Delta L_2}{L_1+L_2}}{\Delta t} \quad (1)$$

where V is the local filament velocity. With the assumption of constant volume, $A_1\Delta L_1+A_2\Delta L_2 = 0$. On the other hand, $A_1 = D_N A_2$, and $\Delta L_2 =$ - $D_N\ \Delta L_1$, where D_N is the natural draw ratio of the neck. Then

$$\frac{dV}{dx} = \frac{\frac{\Delta L_1 - D_N\Delta L_1}{L_1+L_2}}{\Delta t} = \frac{\frac{\Delta L_1(1-D_N)}{L_1+L_2}}{\Delta t} \quad (2)$$

The overall neck propagation speed can be defined either by $\Delta L_1/\Delta t$ or $\Delta L_2/\Delta t$, where the former is for the view of the neck propagation from the undrawn part, and the latter from the drawn part. On the other hand, the number density of neck ρ_N can be defined using the total number of neck, i.e. the portion with step-wise change of diameter, in the region, k, as follows:

$$\rho N = k/(L1 + L2) \quad (3)$$

Then, the propagation speed of individual neck can be expressed either by$\Delta L_1/(k\Delta t)$ or $\Delta L_2/(k\Delta t)$. Since

$$\frac{dV}{dx} = \frac{\frac{\Delta L_1(1-D_N)}{k/\rho_N}}{\Delta t} = \frac{\Delta L_1}{k\Delta t}(1-D_N)\rho_N \quad (4)$$

The propagation speed of individual neck is:

$$\frac{\Delta L_1}{k\Delta t} = \frac{1}{(1-D_N)\rho_N}\frac{dV}{dx} \quad (5)$$

or

$$\frac{\Delta L_2}{k\Delta t} = \frac{-D_N}{(1-D_N)\rho_N}\frac{dV}{dx} \quad (5')$$

The propagation speed of individual neck estimated using equation 5′ versus observation position for various drawing conditions are shown in Fig. 6. A high degree of variance within a similar range was observed for all drawing conditions.

The reason for the high variance of the neck propagation speed is that all the necks are not distributed homogeneously throughout the filaments, and a large number of necks are concentrated in specific locations, while some sections had only a few necks. Therefore, the tendency for the effect of feed roller speed did not appear to be significant but showed a slight increase with the increase of feed roller speed. On the other hand, it was obvious that the neck propagation speed increased with the increase of machine draw ratio regardless of the drawing speed and the observation position.

It should be noted that the propagation speed of individual neck is expected to increase with the increase of local strain rate, and with the decrease of the number density of neck. With the increase of distance

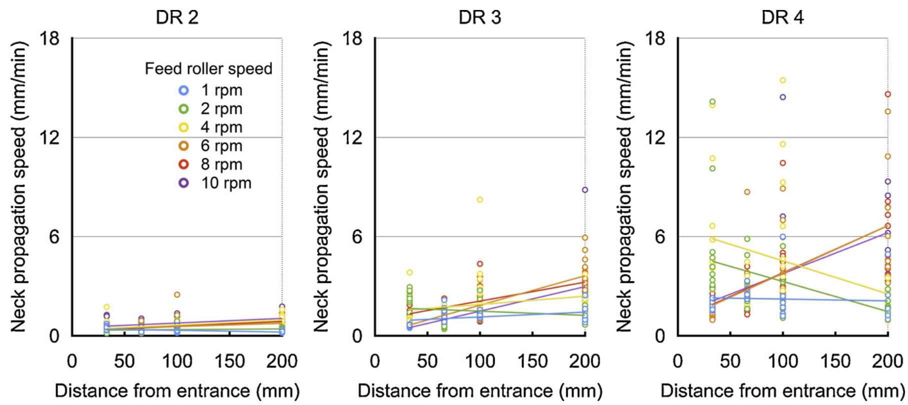

Fig. 6. Variation of the propagation speed of individual neck with the increase of distance from the entrance of ethanol channel without snubber pin. The trend lines were obtained by the least square method for different feed roller speeds. Results for the draw ratios of 2, 3 and 4 are shown.

from the entrance of ethanol channel, it appears that these two factors cancel each other, and eventually any particular trend for the variation of neck propagation speed with the distance from the entrance of ethanol channel could be observed.

3.6. *Effect of snubber pin*

A snubber pin was placed in the ethanol bath to stabilize the tension and induce the initiation of neck. Schematic illustration of the continuous drawing system equipped with a snubber pin in the ethanol bath is shown in Fig. 1(b). Variation of drawing line tension with time for the feed roller speed of 1 rpm and machine draw ratios of 2, 3 and 4 are compared in Fig. 7. Along with the lowering of the drawing tension, stabilization of the tension fluctuation was also observed. Captured images of the drawing line for the drawings with and without pin installation are compared in Fig. 8. It was found that there was enormous increase in the number density of neck after the installation of a snubber pin.

After the pin installation, increase of drawing line tension from the upstream to the downstream of the pin was expected to occur because of the effect of friction force. Therefore, the tension was also measured at the location between the feed roller and the ethanol bath in addition to that between the ethanol bath and the take-up roller. As shown in Fig. 9, the drawing tension at the upstream was significantly lower. Nevertheless, the tension on both sides around the pin were lower than the tension without the pin installation for all feed roller speeds. Stabilization of tension fluctuation with the installation of the pin was also confirmed. Based on these observations, it was speculated that the initiation of neck is the major source for the fluctuation of the drawing line tension, and the installation of a pin may initiate the stable and continuous formation of many necks.

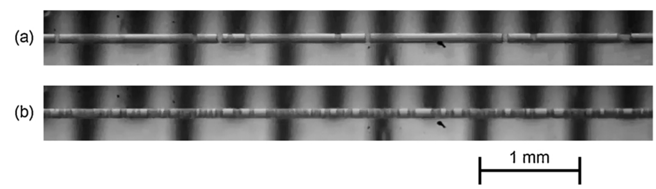

Fig. 8. Photographs for comparing the change in number of necks according to pin installation. (a) is when a pin is not installed and (b) is when a pin is installed. Distance from the entrance of ethanol chancel at the left-end in the photograph is 20 mm, draw ratio is 2, and feed roller speed is 1 rpm.

3.7. *Analysis of infusion behavior using dye*

In order to confirm the infusion behavior during the continuous drawing process, drawing was performed using dye-ethanol solution. Our previous research on the batch-type drawing of PET in dye-ethanol solution suggested that during drawing in ethanol, infusion of solvent occurs at the same time as the propagation of the neck. Furthermore, the solute (dye molecule) is also transported into fiber simultaneously with the solvent [17–19].

In order to compare the infusion efficiency, dyeing was carried out while drawing at various conditions (rotational speed of the feed roller of 1 rpm and 10 rpm, DR 4, with and without installation of pin) to obtain dyed PET filament samples. The concentrations of dyes in the filament were measured and compared by laser Raman spectroscopy. Typical examples of the laser Raman spectra of PET filaments with and without dyeing are compared in Fig. 10. The spectrum of dye is also

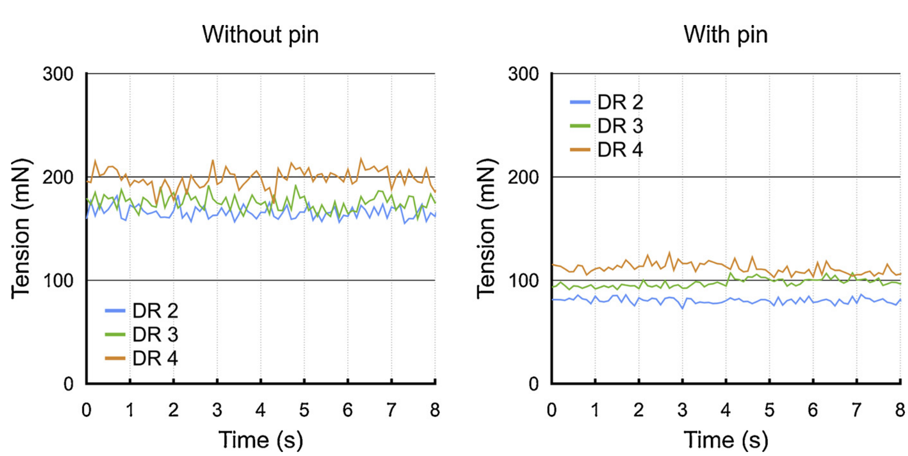

Fig. 7. Result of tension on drawing line with and without the pin installation.

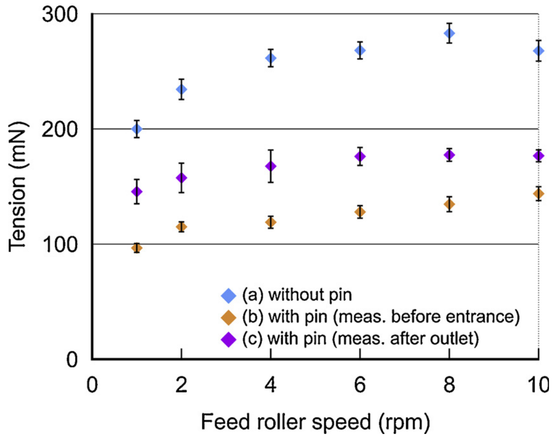

Fig. 9. Impact of snubber pin on draw line tension. (a) is when a pin was not installed and (b) is with snubber pin and measured between feed roller and entrance of ethanol channel (c) is with snubber pin and measured between outlet of ethanol channel and take-up roller. Draw ratio is 4.

shown to indicate the characteristic peaks. As a method of comparing the concentration of the dyes, the intensity of spectra of each PET fiber samples was normalized based on the intensity of 630 cm^{-1} peak corresponding to benzene ring of PET. And then the intensity of 1340 cm^{-1} peak, corresponding to the peak of the dye, was compared.

Fig. 11 shows the result of the evaluation of dye concentration. Since the drawing was performed with the machine draw ratio lower than the natural draw ratio, drawn part and undrawn part co-existed in the drawn fibers. It was confirmed that no infusion of dye occurred for the undrawn part of the filament. Regarding the dye concentration in the drawn part of the filament, concentration was significantly higher for the fibers drawn with the installation of a snubber pin.

In case of the drawing with a pin, even though drawing tension was lower, which may have a negative effect for the infusion, propagation speed of individual neck can be much slower because of a large number of necks. It can be speculated that the slow neck propagation speed may have promoted the infusion of ethanol and dye molecules into the PET filament. On the other hand, increase in the rotation speed of feed roller did not cause much reduction of infusion. This result roughly coincided with the results on the analysis of neck propagation speed shown in Fig. 5. It should be noted that only a limited effect of processing speed on the infusion behavior is a positive characteristics from the view point of the industrialization of this process.

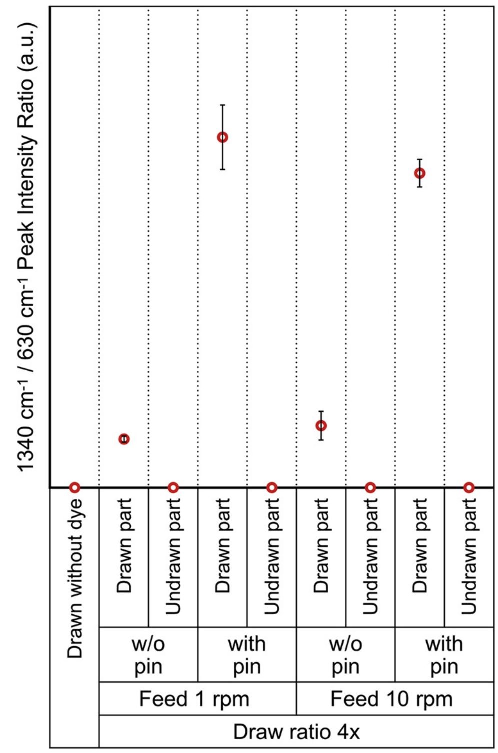

Fig. 11. Concentration of dye in the filament analyzed using laser Raman spectra. Since the draw ratio is lower than the natural draw ratio, drawn and undrawn parts coexist in a single filament after the drawing.

4. Conclusions

As a result of the studies on the continuous drawing process of PET using organic solvent, the occurrence of multiple-neck was observed. The instantaneous draw ratio in the ethanol channel showed higher increase rate at an early stage in the cases of slower drawing speed and/or higher machine draw ratio. At higher drawing speed, higher number of necks initiated, and the number density of the neck decreased as the drawing proceeded toward downstream regardless of the drawing conditions. The neck propagation speed increased as the draw ratio increased, and it was confirmed that the neck propagation speed was influenced more by the draw ratio than the drawing speed. Draw tension increased as the drawing speed increased or draw ratio increased. The stability of the drawing process was improved in presence of a pin

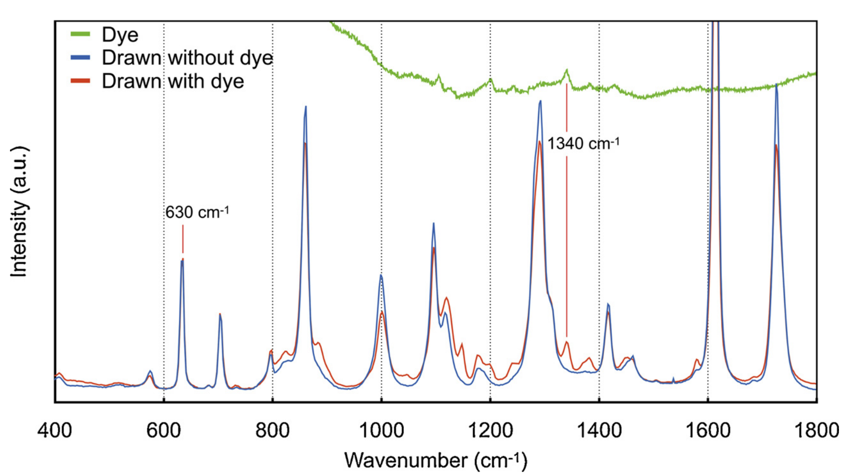

Fig. 10. Comparison of laser Raman spectra of PET filaments with and without dye. The spectra of dye is also shown for comparison. In the spectrum of the filament with dye, additional scattering peak can be observed at 1340 cm^{-1} which correspond to the peak from the dye.

for inducing the necks. It was confirmed that solvent infusion and also solute imbibition occurred in the continuous drawing process simultaneously, and the efficiency of infusion was significantly increased for the process using a pin.

As a conclusion, it was confirmed that the drawing of amorphous and unoriented PET fiber with the infusion of ethanol as well as with the imbibition of dye molecule is possible in the continuous drawing process. This finding suggested the possibility of developing a technology for producing dyed and/or functionalized PET fibers with highly developed fiber structure in a single-step process at room temperature, i.e. without applying heating. Analyses on the relation between processing conditions and structure and properties of the drawn fibers are in progress. Increases of processing speed as well as draw ratio are the research subjects which need to be explored in the future.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

References

[1] I.M. Ward, Structure and properties of oriented polymers, Springer Sci. Bus. Media (2012) 19.
[2] H.M. Heuvel, R. Huisman, Effect of winding speed on the physical structure of As-Spun poly(ethylene terephthalate) fibers, including orientation-induced crystallization, J. Appl. Polym. Sci. 22 (1978) 2229–2243.
[3] T. Kunugi, A. Suzuki, M. Hashimoto, Preparation of high-modulus and high-strength poly(ethylene terephthalate) Fiber by zone annealing", J. Appl. Polym. Sci. 26 (1981) 213–221.
[4] H.H. George, A. Holt, A. Buckley, A study of structural development in the high speed spinning of poly(Ethylene terephthalate)", Polym. Eng. Sci. 23 (2) (1983) 95–99 Mid-February.
[5] M. Masuda, W. Takarada, T. Kikutani, Effect of the control of polymer flow in the vicinity of spinning nozzle on mechanical properties of poly(ethylene terephthalate) fibers, Intern. Polym. Process. XXV 2 (2010) 159–169.
[6] W.R. Moore, R.P. Sheldon, The crystallization of polyethylene terephthalate by organic liquids, Polymer 2 (1961) 315–321.
[7] R.P. Sheldon, A kinetic study of the benzene-induced crystallization of polyethylene terephthalate, Polymer 3 (1962) 27–34.
[8] R.P. Sheldon, P.R. Blakey, Liquid-induced crystallization in polymers, Nature 195 (1962) 172–173.
[9] H. Ouyang, W. Lee, S. Shiue, T. Lin, Solvent-induced crystallization in poly(ethylene terephthalate) during mass transport, J. Polym. Sci. Part B Polym. Phys. 40 (2002) 1444–1453.
[10] Y. Yamashita, K. Monobe, On the cold crystallization of poly(ethylene terephthalate) from its glassy state, Sen'i Gakkaishi 37 (3) (1981) 29–34.
[11] A.K. Kulshreshtha, A.H. Khan, G.L. Madan, X-ray diffraction study of solvent-induced crystallization in polyester filament, Polymer 19 (July) (1978) 819–823.
[12] H. Ouyang, W. Lee, W. Ouyang, S. Shiue, T. Wu, Solvent-induced crystallization in poly(ethylene terephthalate) during mass transport: mechanism and boundary condition, Macromolecules 37 (2004) 7719–7723.
[13] H. Ouyang, C. Chen, Acetone transport in poly(ethylene terephthalate), J. Appl. Phys. 81 (1997) 6680–6683.
[14] H. Takeda, S. Choi, Solvent-induced crystallization of amorphous poly(ethylene terephthalate) Fiber in chlorine substituted hydrocarbons, Sen'i Gakkaishi 48 (9) (1992) 447–453.
[15] A.L. Volynskii, N.F. Bakeev, Solvent Crazing of Polymers (Studies in Polymer Science) vol. 13, Elsevier Science Ltd., Amsterdam, 1995.
[16] O.V. Arzhakova, A.A. Dolgova, L.M. Yarysheva, A.L. Volynskii, N.F. Bakeev, Specific features of the environmental crazing of poly(ethylene terephthalate) fibers, Polymer 56 (2015) 256–262.
[17] R. Khanum, W. Takarada, A. Aneja, T. Kikutani, Crystallization of poly(ethylene terephthalate) filaments by infusion of ethanol upon cold drawing, Polymer 59 (2015) 26–34.
[18] R. Khanum, D. Go, W. Takarada, A. Aneja, T. Kikutani, Effect of drawing speed on infusion and drawing behaviors of poly(ethylene terephthalate) filaments upon cold drawing in ethanol, Sen'i Gakkaishi 71 (9) (2015) 273–283.
[19] R. Khanum, D. Go, W. Takarada, A. Aneja, T. Kikutani, Role of ethanol concentration on drawing and infusion behavior of poly(ethylene terephthalate) filaments upon cold drawing, Polymer 70 (2015) 19–29.
[20] E.G. Rukhlya, O.V. Arzhakova, L.M. Yarysheva, A.L. Volynskii, N.F. Bakeev, Oligomer-polymer blends based on solvent-crazed polymers, Polym. Sci. Ser. B 49 (5-6) (2007) 118–122.
[21] E.G. Rukhlya, E.A. Litmanovich, A.I. Dolinnyi, L.M. Yarysheva, A.L. Volynskii, N.F. Bakeev, Penetration of poly(ethylene oxide) into the nanoporous structure of the solvent-crazed poly(ethylene terephthalate) films, Macromolecules 44 (2011) 5262–5267.
[22] E.G. Rukhlya, L.M. Yarysheva, A.L. Volynskii, N.F. Bakeev, Effects of tensile strain on the peculiarities of PEO penetration into the nanoporous structure of PET deformed via the crazing mechanism, J. Chem. Soc. Faraday Trans. 18 (2016) 9396–9404.
[23] A.P. Andronova, S.I. Popryadukhina, I.A. Egorov, N.B. Aleshicheva, Dyeing polyester fibers using the crazing method, Fibre Chem. 43 (1)July) (2011) 86–89.
[24] R.D. Kale, A. Banerjee, G. Katre, Dyeing of polyester and polyamide at low temperature using solvent crazing technique, Fibers Polym. 16 (1) (2015) 54–61.