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Thesis Outline

Simultaneous Structure Formation and Functionalization of Poly(ethylene terephthalate) Fibers through Infusion of Organic Solvent in Continuous Drawing Process

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This thesis is entitled “Simultaneous Structure Formation and Functionalization of Poly(ethylene terephthalate) Fibers through Infusion of Organic Solvent in Continuous Drawing Process” and consists of five Chapters.

Chapter 1, "Introduction", introduces the fiber formation behavior during drawing of un-oriented amorphous poly(ethylene terephthalate) (PET) fiber in organic solvent, in which the absorption of organic solvent as well as dissolved solute, which is designated as “infusion”, proceeds along with the simultaneous formation of a large number of necks. The infused solvent causes the solvent-induced crystallization, and eventually, the fiber structure formation and functionalization of PET fibers can be achieved in a single-step process at room temperature. Previous studies on the drawing of fibers in organic solvent, however, was performed mainly utilizing the batch type process. Considering the above and with the aim of elucidating the applicability of this process in the industry, the research on the detailed analysis of fiber formation behavior during the continuous drawing of PET fiber in organic solvent is proposed.

In Chapter 2, “In-situ Observation of Drawing Behavior in Continuous Drawing Process of PET Fiber in Organic Solvent”, in-situ observation of the fiber formation behavior during the continuous drawing of PET in ethanol is reported. The continuous drawing was carried out using a set of rollers operating at different rotation speeds and an ethanol flowing-in glass channel placed between the two rollers. Observation using a high-speed camera equipped with a high magnification lenses confirmed the occurrence of multiple-necking. The propagation speed of individual neck, which is considered as a main parameter controlling the infusion behavior, was calculated from the local number density of neck and the gradient of local instantaneous draw ratio. It was confirmed that the neck propagation speed increases as the draw ratio increases, whereas the drawing speed does not show significant influence on the neck propagation speed. It is also reported that the stability of the drawing process can be improved by providing a snubber pin in the ethanol channel. The pin installation resulted in the reduction of drawing stress, suppression of the non-uniform formation of multiple necks with a remarkable increase in the number of necks, and the stable occurrence of necking in the ethanol bath up to the natural draw ratio of the PET fiber.

In Chapter 3, "Infusion Behavior of Organic Solvent in Continuous Drawing Process of PET Fiber", the infusion behavior in continuous drawing of PET fiber in ethanol was investigated applying the differential scanning calorimetry (DSC) and the laser Raman spectroscopy measurements, in which infusion behavior of ethanol and dissolved dye molecules, respectively, were analyzed. In the analysis on the variation of DSC thermograms of PET fibers after drawing, the endothermic peak due to the evaporation of ethanol confirmed the existence of ethanol in the fiber for about one day followed by the gradual reduction of the amount of infused ethanol to almost zero after about one week. From the laser Raman spectroscopic measurement, it was also clarified that the efficiency of dye infusion was higher when the neck propagation speed was lower. Sufficient infusion of dye molecules into the core part of the fiber was also confirmed.

In Chapter 4, "Development of Higher-order Structure and Improvement of Mechanical Properties in Continuous Drawing Process of PET Fiber in Organic Solvent", developments of higher-order structure and mechanical properties of the PET fibers drawn by continuous drawing process using ethanol were investigated in detail. It was confirmed that the ethanol drawn PET fibers have highly-oriented crystalline structure, indicating the occurrence of the ethanol-induced crystallization behavior. Numerous voids were also found to be generated through the drawing in ethanol, and the shape of the voids changed into needle-like shape oriented along the fiber axis with the increase of draw ratio. Analysis of the mechanical properties of drawn fibers revealed the continuous increase of yield stress for about one month after fiber formation. It was also confirmed that the elongation at break of the PET fibers drawn in ethanol was remarkably increased as compared with those drawn without using ethanol. With the intention of producing high-strength PET fibers, the as-spun PET fiber was firstly drawn in ethanol up to the natural draw ratio, and the secondary drawing process was applied in the air for further drawing and annealing. Analyses of the higher-order structure revealed that the crystalline structure did not show any significant difference between the fibers of multi-stage ethanol drawing and conventional drawing. On the other hand, the degree of orientation in core part of the fiber was lower for the fibers drawn in ethanol, whereas the residual stress in the core part of the fiber analyzed applying the laser Raman spectroscopy was more homogenous for the multi-stage ethanol drawn fibers. Nonetheless, it was possible to obtain fibers with a tensile strength of 1.2 GPa through the drawing process using ethanol, where the tensile strength was comparable while the toughness was improved in comparison with the fibers produced through the conventional thermal drawing process.

Chapter 5, "Concluding Remarks", summarizes the results and achievements of this study.