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Thesis Outline

This thesis is entitled “Solvent Infusion Behavior and Structural Development of Poly(ethylene terephthalate) Filament upon Cold Drawing”, written in English and consisting of seven Chapters.

In Chapter 1, “General Introduction”, it is stated that the aim of this thesis is to clarify the behaviors of sucking-in (infusion) of surrounding solvent and higher-order structure development occurring during the drawing of undrawn poly(ethylene terephthalate) (PET) filament in solvent at room temperature.

In Chapter 2, “Crystallization of Poly(ethylene terephthalate) Filaments by Infusion of Ethanol upon Cold Drawing”, drawing of PET filament in ethanol is introduced. In comparison with the ordinary cold drawing in the air or water, where deformation proceeds with the propagation of single neck, it was recognized that the drawing in ethanol caused multiple necks containing undrawn and drawn parts on filament. Change of drawing media from air or water to ethanol also caused a great reduction of yield and drawing stresses as well as a significant increase of natural draw ratio (neck draw ratio). Analysis of drawn filaments of various draw ratios revealed the presence of ethanol in the drawn filament. The amount of infused ethanol increased with the increase of draw ratio, and the development of highly oriented PET crystals was confirmed even though drawing was performed at room temperature. Solvent induced crystallization occurred only when PET filament was drawn in

ethanol, whereas undrawn filament or air drawn filament treated with ethanol did not show any crystallization. In addition, distinct scattering lobe was observed in the small-angle X-ray scattering (SAXS) pattern. It was speculated that formation of multiple neck is due to the reductions of surface free energy and energy barrier for producing new surface through the formation of multiple neck. Based on the fact that the Poisson's ratio of PET filament is less than 0.5, it can be considered that during drawing negative pressure is created in filament by volume expansion and therefore ethanol can be infused. Ethanol can create further opening in polymer structure which causes SAXS scattering. When drawing was performed in the ethanol/dye solution, color was observed only in the drawn parts of filament while the undrawn parts showed no color. This result indicates that solute also can be infused in filament upon cold drawing along with the infusion of solvent.

In Chapter 3, "Role of Ethanol Concentration on Drawing and Infusion Behavior of Poly(ethylene terephthalate) Filaments upon Cold Drawing", effect of ethanol concentration of ethanol water solution on infusion and drawing behavior of PET filament was investigated. It was recognized that the increasing of ethanol concentration caused the increasing of the number of necks on filament. This result supported the consideration that the formation of multiple necks is due to the reduction of surface free energy. With increasing of ethanol concentration, yield and drawing stresses reduced dramatically while natural draw ratio increased. Amount of ethanol infused into filament also increased with the ethanol concentration, which accompanied the higher crystallinity, stronger equatorial peak in SAXS and higher amount of infused dye. Cold crystallization temperature was found to decrease with increasing ethanol concentration indicating that even the characteristics of undrawn part

can be altered during the drawing in ethanol presumably because of the effect of applied tensile stress. It was also discussed that the sufficient amount of infusion at lowered ethanol concentration is an important finding from the view point of safety for the industrialization of this process.

In Chapter 4, “Effect of Drawing Speed on Infusion and Drawing Behavior of Poly(ethylene terephthalate) Filaments upon Cold Drawing in Ethanol”, the effect of drawing speeds on infusion and drawing behavior of filament was investigated. It was found that the increasing of drawing speed caused the increasing of yield and drawing stresses as well as the increasing of number of neck. Propagation speed of individual neck also increased with the increasing of drawing speed. Number of neck was also found to decrease with the increasing of the period of immersion of filament in the solvent before starting the drawing. On the other hand, amount of ethanol infused into filament increased with the decreasing of drawing speed, which caused the increasing of crystallinity and equatorial scattering intensity in SAXS as well as the increasing of amount of dye infusion into filament. These results suggested that a certain kinetic mechanism is also involved for the neck appearance which has been discussed based on the variation of surface free energy. It was also discussed that the time required for the infusion of ethanol into filament through neck deformation is comparable with the drawing speed applied in this experiment.

In Chapter 5, “Removal of Infused Ethanol from Drawn Poly(ethylene terephthalate) Filaments”, the possible ways for removing infused ethanol from drawn filaments were discussed. After water washing endothermic peak in the differential scanning calorimetry thermograms caused by the infused ethanol disappeared completely, while there was no great

change in color and crystalline structure in filament. Water washed away only ethanol but dye molecules stayed in filament. On the other hand, equatorial scattering intensity of SAXS patterns almost vanished after water washing indicating that the needle-like void structure or swelled amorphous structure can be formed only when ethanol exists in the filament. It was also found to be possible to remove infused ethanol from drawn filament by applying additional drawing in the air.

In Chapter 6, “Infusion and Structural Development of Partially and Low Oriented Poly(ethylene terephthalate) Filaments Drawn in Solvent”, infusion behavior of partially oriented PET filament was investigated. Partially oriented filament did not show multiple necks during drawing in ethanol, and merely a small amount of infusion was detected. Drawn filament prepared by drawing the partially oriented filament in ethanol showed crystalline reflection but less distinct than those prepared from low oriented filaments.

In Chapter 7, “General Conclusion”, results obtained in each Chapter is summarized through discussing the mechanism for the up-take of solvent through infusion and structure formation during the cold drawing of undrawn PET filament in solvent. Furthermore, possibility for the industrialization of this process is discussed based on the fundamental knowledge acquired through this research.