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題目(和文)	Fabrication of All-thermoplastic Fiber-reinforced Composites through Compression Molding of High-speed Spun Bicomponent Fibers
Title(English)	高速紡糸複合繊維の圧縮成形による全熱可塑性樹脂繊維強化複合材料の作製
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Thesis Outline

A technology for preparing all thermoplastic fiber reinforced composites through the compression molding of sheath-core bicomponent fibers was studied. In this system, the core component of the sheath-core bicomponent fiber acts as the reinforcing fiber whereas the sheath component acts as the matrix polymer after the compression molding. Advantage of this technology is to overcome the difficulty of impregnating reinforcing fibers into thermoplastic matrix. Properties of the final products are dependent on various critical step for the production of composites. The mechanical properties of fiber-reinforced composites are naturally influenced strongly on the mechanical properties of sheath-core bicomponent fibers, which were produced through the high-speed melt spinning process to introduce well-developed fiber structure to the core component.

In chapter 2, spinning behavior of fibers in spunbonding process was analyzed because the process can be utilized for the direct fabrication of fiber web. Fiber-reinforced composites with two-dimensional random fiber orientation can be prepared through the compression molding of the web if it is consist of sheath-core bicomponent fibers. In addition, fiber structure development occurs during the spinning process and the structure and properties of the as-spun fibers are mostly determined by the thermal and stress histories the polymer melt experiences in the spin-line. From the results of spin-line velocity measurement of single component poly(ethylene terephthalate) (PET) fibers, it was found that fiber speed increased with the increase of air pressure and also by changing the position of air-ejector closer to the spinneret, while with the reduction of distance between spinneret and the air-ejector, there appeared an inflection point in the fiber velocity versus distance relation. From the wide-angle X-ray diffraction (WAXD) analysis of the as-spun PET fibers, orientation-induced crystallization was found to occur when the spinning velocity exceeded 4 km/min. It was also found that the starting of the orientation-induced crystallization corresponds to the appearance of the inflection point. Numerical simulation of the spunbonding process was carried out introducing the effect of orientation-induced crystallization. The air-friction force applied by the air-ejector was estimated based on the parameters acquired through the off-line tension measurement for the static fibers of various diameters in the air-ejector. It was possible to reproduce the upward shift of the solidification position accompanied by the increase of solidification temperature of the spin-line from the glass transition temperature to crystallization temperature, which increased with the increase of fiber velocity. The inflection point also could be reproduced in the simulation. It was found that the appearance of the inflection point originates from the additional effect of the air-friction force caused by the shift of solidification temperature.

In chapter 3, fiber reinforced composites were prepared through the compression molding of sheath-core bicomponent fibers consisting of PET (core)/ poly(butylene terephthalate) (PBT) (sheath) and thermoplastic liquid crystalline polymer (TLCP) (core)/ PET (sheath). Ordinary high-speed melt spinnig process with the use of a take-up winder and spunbonding process with the use of an air-ejector were applied for the preparation of the fiber bundle

and the web consisting of randomly oriented fibers, respectively. Properties of the composites is expected to depend strongly on the state of orientation of fibers as well as the properties of the core part of the bicomponent fibers, which is determined mostly by the thermal and stress histories applied to the molten polymer in the spin-line. In the spunbonding process, fiber speed increased with the increase of air pressure and the change of air-ejector position closer to the spinneret as expected, while it was found that the fiber speed became lower and change of fiber speed with the change of air-ejector position was less significant when viscosity of polymer is high. Structure development of the as-spun sheath-core fibers was investigated through the analyses of birefringence and WAXD diagram. It was found that in the PET/PBT bicomponent fibers, structure development of PET was enhanced while that of PBT was suppressed in comparison with the single component spinning of individual polymers. In the case of TLCP/PET, well developed fiber structure was observed for the TLCP component while PET exhibited the amorphous structure of low orientation at all the take-up velocities investigated. In other words, in the high-speed spinnig of bicomponent fibers for the preparation of fiber-reinforced composites, the structure development of the core and sheath components, which will be the reinforcing fiber and the matrix polymer after the fabrication of composites through the compression molding, tended to be enhanced and suppressed respectively, in comparison with those for the individual single component spinnings. This tendency arises from the requirement of the selection of the combination of core and sheath components with the core component having the nature of solidification at higher temperatures through the crystallization in the process. Fiber reinforced composites with uni-axial and random fiber orientation were prepared directly through the compression molding of bicomponent fibers. In case of PET/PBT, appropriate compression molding temperature was between the melting temperatures of PET and PBT, while in case of TLCP/PET compression molding temperature of 180 °C was applicable because PET component in the sheath was in an amorphous state. In both cases, oriented crystalline structure of core components was maintained after the compression molding, and the composites with fairly high mechanical properties could be prepared. Theoretical prediction of the mechanical properties of composites with random fiber orientation yielded the values which have good agreement with the experimental results.

In chapter 4, fiber-reinforced single-polymer composites of PET were fabricated successfully through the compression molding of the high-speed melt-spun sheath-core bicomponent fibers consisting of high molecular weight PET (HMPET) and low molecular weight PET (LMPET) as the core and sheath components, respectively. When the take-up velocity exceeded 3 km/min, orientation-induced crystallization started to occur in the HMPET component while the LMPET component remained in an amorphous state and its birefringence decreased with the increase of take-up velocity because of the orientation relaxation after the starting of the crystallization of HMPET in the spinning process. Compression molding of the sheath-core fibers with highly oriented and crystallized core component and amorphous and low oriented sheath component was conducted at 180 °C, the temperature between the glass transition and melting temperatures of PET. At this temperature, rubbery softening of the sheath component was utilized for the adhesion of sheath component to form matrix phase in the composite while maintaining the well-developed fiber structure of the core component intact. The fabricated fiber-reinforced single-polymer composites prepared from the bicomponent fibers obtained at 5 km/min exhibited fairly high flexural strength and modulus of 144 MPa and 3.89 GPa, respectively, in the case composites with uni-axial fiber orientation. The composites of random fiber

orientation prepared from the compression molding fiber web manufactured through the spunbonding process also exhibited fairly high mechanical properties. It was emphasized that the fabricated fiber-reinforced single-polymer composites were consisting of pure PET without any chemical modification such as copolymerization. The sheath and core components merely have different molecular weights. When the fabricated composite is melted, the molecular weight distribution is expected to be the theoretical value of Mw/Mn = 2 because of the transesterification. This means that the recyclability of the fabricated composites is exceptionally high even in comparison with other fiber-reinforced all-thermoplastic single-polymer composites.

Conditions for fabricating all-thermoplastic fiber-reinforced composites including single-polymer fiber-reinforced composites through the compression molding of high-speed spun bicomponent fibers were investigated in detail in this research. In conclusion, it can be said that the experimental results clarified key factors for preparation of the composites of good mechanical performances through the selection of combination of polymers for bicomponent spinning, the control of structure development behavior of individual components in the high-speed melt spinning process and the selection of appropriate compression molding conditions based on the structure of sheath component.