6A & 9C /2011G-123

SAXS and WAXS Studies on Guest Exchange Processes in Crystalline Complexes of Syndiotactic Polystyrene

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1 Introduction

Syndiotactic polystyrene (sPS) is a relatively new commodity polymer, which exhibits a variety of solid states. One of the important properties of sPS is the formation of co-crystal structures, where organic compounds are stored as guests in the cavities formed between polymer sheets consisting of sPS helices with TTGG conformation, as shown in Figure 1. It has been clarified that a wide range of chemical compounds including functional molecules can be incorporated into sPS lattice, for examples, crown ethers, organic radicals, fluorescent and dye molecules, and even polymeric molecules. The orientation of the guest compound can be adjusted by controlling the arrangement of the crystallites of the clathrate phase. Now sPS crystalline complexes are regarded as of high potential for polymer based composite materials.

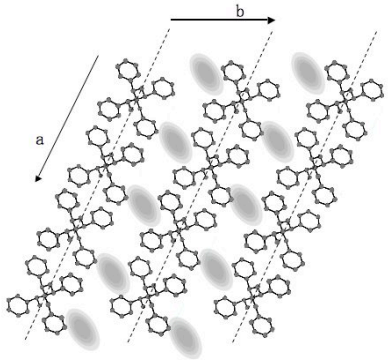

Fig. 1. Crystal structure of sPS complex.

The guest molecules in the sPS complexes can be replaced smoothly by exposing to a vapor or a liquid of another kind of molecule. The guest exchange method has an advantage that enables sPS to form crystalline complexes even with chemical compounds that are difficult to incorporate into the crystalline region by usual solution-cast and solvent-induced crystallization methods.

In order to obtain information how old guests are replaced with new ones, we tried to follow the guest exchange process from chloroform to triethyleneglycol dimethyl ether (TEGDME) by simultaneous WAXS and SAXS measurements.

2 Experiment

sPS was provided by Idemitsu Bussan Corp. In order to measure clear lamellar reflections in a well defined area, 4-5 times uniaxially drawn films about 50 μm thick were prepared. sPS/chloroform complex samples were prepared by exposing the films to Chloroform vapor.

Time resolved SAXS and WAXS measurements were carried out at BL-6A and BL 9C by using an imaging plate for WAXS and a CCD camera system for SAXS measurements.

The guest exchange process was initiated by injecting TEGDME into a glass capillary containing several pieces of sPS/chloroform complex film.

3 Results and Discussion

Figure 2 shows the changes in SAXS profile during guest exchange. The reflection corresponding to the lamellar repeat distance clearly shifts to a lower angle after a 3-hour latent period from the inception of the guest exchange, which indicates that the lamellar spacings are expanded during the course of the guest exchange.

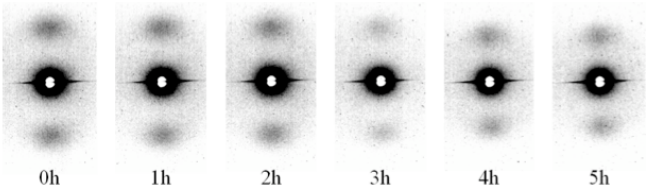

Fig. 2. Changes in SAXS profiles during sPS guest exchange process.

It has been found that the length of the latent period significantly depends on the sample condition as shown in Figure 3. Sample A, which was well dried after the complexation by chloroform, exhibits a long latent time, whereas Sample B, which was subjected to the guest exchange procedure just after the complexation with chloroform, immediately starts the lamellar expansion.

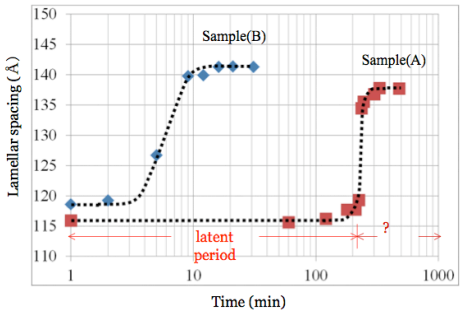

Figure 3. Time dependence of lamellar spacing.

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