Temperature Dependent Atomic Force Microscopic Study of Microphase-separated Structure of Polyurethane Elastomers

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Introduction

Block copolymers form a microphase-separated structure as the result of chain connectivity and the inherent thermodynamic incompatibility between the constituents of the copolymer. Microphase separation of block copolymers results in variety of applications, such as thermoplastic elastomers, compatibilizers, surface modifiers and photoresists. Polyurethane elastomers (PUEs), which are a multi-block copolymer, are widely used as thermoplastic elastomers, and their properties are related to the microphase-separated structure. The purpose of this study is observation of the microphase-separated structure of PUEs using temperature controllable atomic force microscope (AFM).

Experimental

PUE was synthesized from poly(oxypropylene) glycol (PPG), 4,4’-diphenylmethane diisocyanate (MDI) and 1,4-buthane diol (BD) by a prepolymer method. Hard segment content was 24 wt%. The microphase-separated structure of PUE was investigated using AFM at various temperatures.

Results & Discussion

Figure 1 shows AFM phase image of PPG-MDI-BD-based PUE at 27 and 117 °C in vacuo. At 27 °C, isolated darker dots surrounded with a brighter matrix were distinctly observed. Since the areal ratio of the darker dots is almost comparable with the hard segment content, it seems likely to consider that the darker and brighter regions correspond to the crystallized hard segment phase and soft segment one, respectively. With increasing temperature, the spherical hard segment domains were changed to a cylinder-like structure. This behavior implies short-range reorganization within the hard segment domains. Furthermore, the microphase-separated structure disappeared above 163 °C. This suggests melting of the hard segment domains.

![Figure 1](image-url)  
(a) 200 nm  (b) 200 nm

Figure 1. AFM phase image of PPG-MDI-BD-based PUE at (a) 27 and (b) 117 °C in vacuo.