Observation of Polymer Blends by X-Ray Phase Tomography

Atsushi Momose[a)], Akiko Fujii[b)], Yoshio Suzuki[c)], Hidekazu Kadowaki[d)], Yukihiro Nishikawa[d)], and Hiroshi Jinnai[d)]

[a)]*Department of Advanced Materials Science, Graduate School of Frontier Sciences, The University of Tokyo, 5-1-5 Kashiwanoha, Kashiwa, Chiba 277-8561, Japan*
[b)]*Department of Applied Physics, School of Engineering, The University of Tokyo, 5-1-5 Kashiwanoha, Kashiwa, Chiba 277-8561, Japan*
[c)]*JASRI/SPring-8, Mikazuki, Hyogo 6791-5198, Japan*
[d)]*Department of Polymer Science and Engineering, Kyoto Institute of Technology, Matsugasaki, Sakyoku, Kyoto 606-8585, Japan*

Blending polymers is of importance because properties of plastic materials can be tailored for specific applications. Phase separation is observed in polymer blends and plays a role determining properties. Its structural analysis is therefore needed especially three dimensionally. X-ray phase tomography is a candidate for that purpose since its high sensitivity enables us to depict phase-separation structures without treatments, such as labeling with high-Z elements or selective etching, for contrast enhancement.

We demonstrate phase separation in blends of polystyrene (PS) and poly(methyl methacrylate) (PMMA) with phase tomography using a crystal X-ray interferometer. Mixtures of PS and PMMA with 1:1 volume ratio were kept at 180 °C for phase separation in cylindrical holes 2 mm in diameter made on a copper plate sandwiched in a melt-press machine. A sample 2 mm in diameter was thus prepared and put in a cell filled with water placed in a beam path of the X-ray interferometer. Phase measurements were performed with 17.7-keV X-rays at 400 angular positions during 180° sample rotation, and a three-dimensional image mapping the refractive index was reconstructed. A CCD-based X-ray image detector of a 3.14-μm effective pixel size was employed.

As shown in Fig. 1, bicontinuous feature of PS-rich (dark) and PMMA-rich (bright) regions was depicted clearly. The density of each region evaluated from the image is consistent with spinodal decomposition. Thus, phase tomography is a powerful and unique technique for three-dimensional observation of a polymer blend quantitatively.

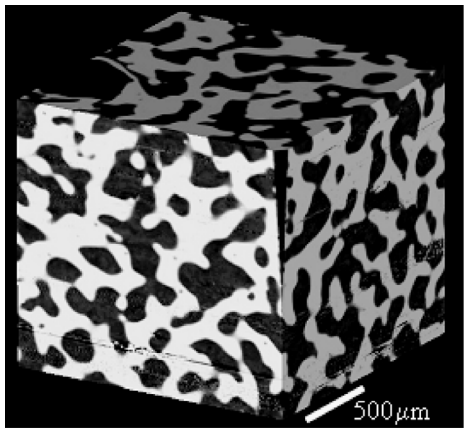

Fig. 1 Three-dimensional image of a PS/PMMA blond obtained by phase tomography.