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In situ GISAXS Investigations of pH and Temperature Responsive Block Copolymer Thin Films during Swelling in Water Vapor

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Responsive block copolymer thin films are of interest for many applications, e.g. as switchable membranes. Many of these systems are based on physical hydrogels, where a hydrophilic midblock is end-capped by hydrophobic end groups. Their dynamics can be tuned by employing temperature responsive polymers as end blocks. In thin films, a pH-responsive midblock may be used to tune the self-assembly process, while the end blocks may be immobilized by temperature variation and the so created nanostructure may be frozen. The latter feature may be exploited in solvent vapor annealing (SVA), which is a technique to improve long-range order and alter the morphology in thin films, but has the drawback that morphologies achieved in the swollen state are often difficult to preserve during solvent removal.
In the present work, thin films from the pentablock terpolymer P(n-BuMA$_8$-co-TEGMA$_8$)-b-PDMAEMA$_{50}$-b-PEG$_{46}$-b-PDMAEMA$_{50}$-b-P(n-BuMA$_8$-co-TEGMA$_8$) are investigated in-situ during SVA using grazing-incidence small-angle X-ray scattering (GISAXS). The films were prepared via spin coating from aqueous solutions of different pH and were subsequently subjected to SVA using water vapor, with experiments being performed at various temperatures. The pH during film preparation is found to play a major role: Films prepared at high pH do not show structural features in the GISAXS maps, even after SVA with water. In contrast, films prepared at low pH feature a well-defined spherical morphology, which is enhanced after SVA.

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