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Characterizing 3-D Assembly of Block Copolymer Films using Rotational Small Angle Neutron Scattering

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Directed Self-Assembly (DSA) using graphoepitaxy, chemically patterned templates, and directed fields has been shown to be an effective route to achieving long range order over short time scales in block copolymer films designed for use as sacrificial resists. However, key questions remain regarding the materials-processing relationship that drives assembly in thin films. Understanding these relationships will become increasingly more important in emerging applications such as in photovoltaics and nanostructured membranes where the polymers are functional components. Utilizing a methodology to sample the entire 3-dimensional fourier space of a thin film coating, termed Rotational Small Angle Neutron Scattering (R-SANS), our group has discovered a complex process window that drives orientation and order in thin film block copolymer assembly. Data from R-SANS is complemented by specular neutron reflectivity (NR) and real time measurements using grazing incidence small angle x-ray scattering (GI-SAXS). In this talk, I will highlight some of our recent results that elucidate the effects of solvent content, evaporation rate, and thermal history on the average orientation and orientation distribution in block copolymer films. Of particular interest is the previously unreported effect of thermal gradients. Using static thermal gradients, we show that the kinetics of assembly are dramatically faster. Specifically, grain sizes on the order of 10s of microns are achievable in minutes for a system that would require >10 h to achieve the same results in a thermally uniform environment. The scaling of kinetics as a function of the static thermal gradient is also discussed and models of the physical processes involved presented. The complex evolution of morphological orientation is then further biased through the application of dynamic gradients in a technique we label as cold zone annealing, a technique suitable for manufacturing methods such as roll-to-roll processing.