TIME-RESOLVED SAXS TECHNIQUE FOR STUDYING BLOCK COPOLYMERS UNDER SHEAR DEFORMATION. S. Suehiro, K. Saji, T. Seto, M. Kakiuchi, T. Hashimoto, Department of Polymer Chemistry, Kyoto University, Kyoto 606-01, Japan, and Y. Amemiya, Photon Factory, National Laboratory for High Energy Physics, Tsukuba 305, Japan.

The investigation of pattern formation in block copolymers has become a fascinating research topic in statistical mechanics of complex fluids in recent years. In situ, time-resolved scattering experiments under shear are essential to understanding the structural mechanisms of shear deformation of block copolymers and other mesostructured materials, owing to the fact that such systems are expected to have numerous kinds of microscopic responses to applied stress.

A synchrotron-radiation dynamic small-angle X-ray scattering (SR-DXSAXS) system has been developed at the Photon Factory, National Laboratory for High Energy Physics, Japan, in collaboration with the Department of Polymer Chemistry, Kyoto University, Japan, utilizing an imaging plate (IP) as a two-dimensional X-ray detecting system, and a hydraulic driving system as a sample deformation device in order to study time-resolved structural changes in polymer specimens, especially block copolymers, subjected to a large oscillatory shear strain.

The specimen is sandwiched between two metal plates and subjected to a large amplitude oscillatory shear strain. The time resolved small-angle X-ray detecting system consists of an IP mounted on a YZ stage driven by a pair of stepping motors and an intelligent stepping motor controller. There is a 100 mm x 100 mm square aperture for the scattered X-rays in front of the IP stage. An IP (400 mm x 200 mm in size) divided into eight sections (100 mm x 100 mm) is used for detecting the SAXS patterns. Each section is moved to the position of the aperture in turn by the two stepping motors to record the X-ray pattern. The time required for the translational movement of the IP stage along the Y-axis or the Z-axis is ca. 0.5 s/100 mm. Therefore, this system can follow the change of structures at a mesoscopic level on a time scale as short as one second. DSAXS studies were performed on polystyrene-block poly(ethylene-alt-propylene) copolymers concerning the dynamic deformation of a body-centered-cubic (bcc) lattice of spherical microdomains, as well as the dynamic orientation and deformation of alternating lamellar microdomains, under a large oscillatory shear deformation.

SCATTERING STUDIES OF FRACTAL SCALING IN POLYMER BLENDS. G. Beaucage*, S. K. Sukumaran, Department of Materials Science and Engineering, University of Cincinnati, Cincinnati, OH 45221-0012.

A fundamental approach to the inclusion of fractal concepts in polymer thermodynamics will be presented. Fractal scaling effects in polymer blends will be demonstrated through small-angle neutron and X-ray data. Fractals offer a natural lexicon for the description of polymeric structures. In spite of this, the incorporation of fractals into thermodynamic models for polymers has been mostly overlooked. We propose that a manifestation of this is the introduction of composition and molecular weight dependencies in the second virial coefficient or the associated Flory interaction parameter as measured by small-angle scattering. We have modified Flory's lattice approach to polymer blends to account for changes in the fractal dimension through the entropy of mixing. This allows for a simple description of global components of entropy in polymer blends and solutions. Our approach is particularly suited to scattering measurements since the mass fractal dimension is directly obtained as the negative of the slope for a power-law regime spanning several decades in q for common polymer blends. In addition to solvent quality, topology, orientation and pressure will shift the fractal dimension in predictable ways. If these deviations of the fractal dimension from the expected values are ignored in thermodynamic approaches, unexpected dependencies of the interaction parameter on composition, molecular weight, topology, strain and even scattering vector can result. Our modified Flory-Huggins approach can be used to describe composition dependencies in the non-combinatorial entropy term of the interaction parameter by comparison with Flory's equation for the free energy. Calculations using this approach agree with previous neutron scattering measurements by Wignall and Bates in PS-dPS. Thus, this approach can be used to correct errors in prior efforts using Gaussian scaling. Moreover, by directly treating scattering data using our approach, unexpected composition and molecular weight dependencies can be removed from the interaction parameter returning it to the simple local site-site parameter for which it was intended.

SMALL ANGLE SCATTERING WITH POLARIZED NEUTRONS: A NEW SPECTROMETER AT ORPHEE. H. Glätti, M. Eiseckremer and C. Ferman, DRECAM/SPEC, CEA Saclay, France, M. Pinot, LLB, CEA-CNRS, France.

A spectrometer for small angle scattering with polarised neutrons has been installed at the ORPHEE reactor. It uses a beam of 8.010 Å deviated from the cold neutron guide G5 by Ni-Ti multilayers and polarized by a supermirror. After a collimation length of 7 m, the neutron intensity at the sample is 1.5.106 n/cm²/s. Due to space limitations, the position sensitive detector is placed outside the vacuum tank. This limits the sample-to-detector distances to discrete 1m-steps from 0.8 to 3.8 m.

The inconvenience of a fixed wavelength and wavelength-spread counterbalanced by a simple and economical design with no need for a velocity selector at a guide end. A superconducting magnet with a homogeneous horizontal field of 3.5 T and a dilution insert which cools a 4He-filled sample cavity to 0.2 K makes this spectrometer particularly suited for studies of spin contrast variation using dynamic nuclear polarization. Examples of polarization-dependent scattering from polymers are shown to demonstrate the power of this method.


We have utilized a “multilayer” monochromator (a pair of layered synthetic microstructures) in small-angle x-ray scattering and diffraction studies of biological materials. Many biological applications of the small angle scattering technique, in particular time-resolved studies, are often limited by the flux incident on the sample. The wider energy bandwidth of the multilayer monochromator can provide a higher beam flux by up to two orders of magnitude as compared with that of the typical Si(111) double-crystal monochromators used at synchrotron beam lines. On Beam Line 4-2 at the Stanford Synchrotron Radiation Laboratory, we have implemented two types of multilayers Mo/C and Mo/B/C in the standard SSSL/SLAC monochromator tank for the studies of x-ray fiber diffraction and solution scattering. In the fiber diffraction experiments, a pair of the latter type of multilayers provided ~10² photons/s in the beam size 0.6 mm x 1.5 mm (FWHM) at photon beam energy 9 keV, an increase of a factor of ~10 over Si(111) in the same configuration. We observed no significant smearing or effects of increased beam divergence due to the wide energy bandwidth in fiber diffraction patterns and low-angle resolution in solution scattering was likewise unaffected. These results demonstrate significant advantages of the multilayer monochromators over Si crystal monochromators for non-crystalline biological x-ray diffraction applications.