Development of In Situ Grazing Incidence Small-/Wide-angle X-ray Scattering for Bulk Heterojunction Thin-film Solar Cells at NSRRC

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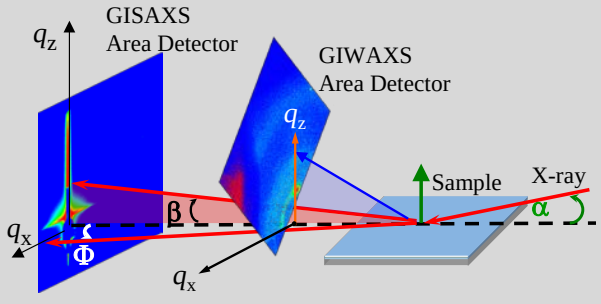
At the National Synchrotron Radiation Research Center (NSRRC), the recent upgrade of the 23A SWAXS endstation with a Pilatus 1M-F (133 Hz) and a Mythen-3K (472 Hz) detectors has advanced synchronized small- and wide-angle X-ray scattering (SAXS/WAXS) measurements for structural kinetics into millisecond resolution. Together with an additional flat panel area detector CMOS-9728DK (3.3 Hz), the integrated detecting system covers a wide-range of X-ray scattering angle with several different modes of operation, including transmission (T), grazing incidence (GI), and anomalous (A) SAXS/WAXS. Particularly, a solvent-vapor controlled chamber with a heating stage has been developed allowing observation of time-resolved, simultaneous grazing incidence SAXS/WAXS (GISAXS/GIWAXS) upon drying, heating, or isothermal annealing of organic thin films for bulk heterojunction (BHJ) photovoltaic solar cells comprising, in general, binary mixtures of conjugate polymers and fullerene derivatives. Crystallization behavior including kinetics (crystalline orientation and size) of the polymer in organic BHJ solar cells can be studied with GIWAXS, whereas aggregation behavior (from several to several tens nm) of the fullerene derivatives and hierarchical large grain size spacing (several hundreds of nm) can be observed with GISAXS. The GISASX/GIWAXS performance is illustrated via a concomitant observation of [6,6]-phenyl-C_{61}-butyric acid methyl ester (PCBM) aggregation and poly(3-hexylthiophene) (P3HT) crystallization in bulk heterojunction (BHJ) thin film (ca. 85 nm) solar cells. With a time and spatial resolutions (5 s/frame; minimum $q \approx 0.004$ Å^{-1}), synchrotron GISAXS has captured in detail the fast growth in size of PCBM aggregates from 7 to 18 nm within 100 s of annealing at 150 °C. Simultaneously observed is the enhanced crystallization of P3HT into lamellae oriented mainly perpendicular but also parallel to the substrate. An Avrami analysis of the observed structural evolution indicates that the faster PCBM aggregation follows a diffusion-controlled growth process, whereas the slower development of crystalline P3HT nanograins is characterized by constant nucleation rate.

Figure 1. Schematic for the simultaneous, grazing incidence small- and wide-angle X-ray scattering at 23A SWAXS beamline of NSRRC.



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