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EXPERIMENTAL METHODS BASED ON GRAZING INCIDENCE AT THE 1W1A BEAMLINE OF THE BEIJING SYNCHROTRON RADIATION FACILITY AND ITS APPLICATION IN CHARACTERIZING THE CONDENSED STATE STRUCTURE OF CONJUGATED POLYMERS

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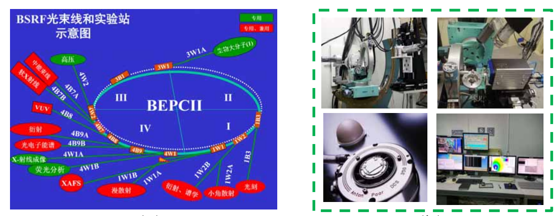
Abstract

The Beijing Synchrotron Radiation Facility (BSRF) 1W1A Diffuse X-Ray Scattering Station can conduct grazing incidence wide/small angle scattering (GIWAXS/GISAXS) experimental method characterization, which is an important method for characterizing the condensed structure of conjugated polymers. To this end, we have upgraded and optimized the experimental method of grazing incidence. After updating the EIGER 1M area detector and reducing stray light interference, the exposure time of a single sample was reduced from 300 seconds to 30 seconds. And we have developed a remote rapid sample change platform, which can achieve remote testing operations outside of the hutch, greatly reducing testing time, and enabling users to remotely conduct online testing operations in their own labs. Subsequently, we further established in-situ steam treatment, in-situ thermal annealing, in-situ drip coating, in-situ spin coating, in-situ scraping coating, and GISAXS testing platforms, enriching the beamline’s grazing incidence methods. In the future, relying on the 1W1A diffuse X-ray scattering station, more in-depth research can be conducted on the crystallization behavior, film formation process, crystallization and phase separation size, and film structure of solution processed conjugated polymers.

INTRODUCTION

The Wiggler insert 1W1 in the storage ring I area of the Beijing Synchrotron Radiation Facility has led out two stations: 1W1A diffuse scattering and 1W1B-XAFS experimental stations (Fig. 1(a)). The 1W1A station utilizes the dual focusing monochromatic X-ray provided by the beam line to conduct structural research on crystals and thin film materials. This station can be operated in both dedicated and parasitic modes. The main optical components of the 1W1A beam line include an asymmetric cut crystal monochromator and a vertical bent reflector. The asymmetric cut crystal monochromator is 19 meters away from the light source, achieve monochromatization and horizontal focusing of the beam, and splitting with the 1W1B beam. The vertical bent reflector is located 1.6 meters behind the monochromator, used to realize vertical focusing of the beam and effectively suppress high-order harmonics. This beamline can conduct experiments such as high-resolution diffraction (XRD), low angle reflection (XRR), grazing incidence diffraction (GIXRD), GIWAXS, and GISAXS.



(a) (b)

Figure 1: (a) Schematic diagram of BSRF Beamline Station; (b) Multiple experimental platforms of the 1W1A.

Recently, we optimized the detectors, flight channels, attenuators, beamstop, etc., and reduced the exposure time of a single sample from 300 seconds to about 30 seconds. And we have developed a remote rapid sample change platform for grazing incidence experiments. Subsequently, we further established a series of in-situ steam treatment, in-situ thermal annealing, in-situ drip coating, in-situ spin coating, in-situ scraping coating, and GISAXS testing platforms, enriching the grazing incidence experimental methods (Fig. 1(b)).

REMOTE OPERATION GRAZING INCIDENCE EXPERIMENTAL PLATFORM

This experimental platform is equipped with a fast sample change device, which saves time for calibration of the sample in grazing incidence mode. Paired with E63 intelligent lightweight 6-degree of freedom modular collaborative robot, it can achieve continuous sampling without entering the hutch, and can achieve continuous testing of hundreds of samples (Fig. 2). Utilization of remote control software, users can realize remote operation of the experiment, greatly reducing the testing time for conventional thin film samples and simplifying the testing steps. (`http://202.122.38.138/docs/1w1aremote.mp4`).

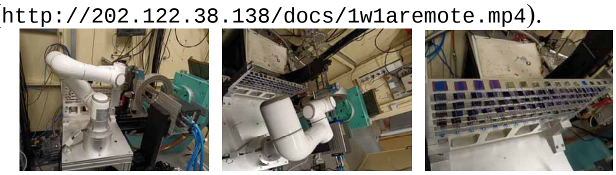


Figure 2: Remote operation GIWAXS platform.

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The crystallization behaviour of solution processing conjugated polymers is a key factor determining their condensed state structure and device performance. To meet user's needs, 1W1A station has established in-situ steam treatment, in-situ thermal annealing, in-situ drip coating, in-situ spin coating, and in-situ scraping coating experimental platforms, which can conduct in-depth research on the crystallization behaviour and film formation process of solution processing conjugated polymers (Fig. 3).

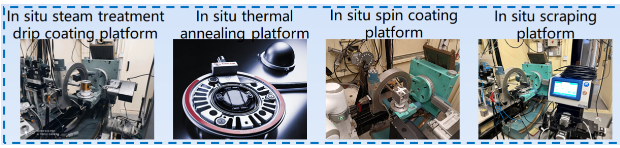

Figure 3: In-situ platform based on conjugate polymerization crystallization behavior and film formation process.

RECENT EXPERIMENTAL RESULTS

Prof. Yanchun Han (Changchun Institute of Applied Chemistry, Chinese Academy of Sciences) and Dr. Hongxiang Li (Sichuan University), have systematically studied the condensed structure and crystallization behavior of conjugated polymers and published related papers (Fig. 4) [1-5]. The in-situ GIWAXS mode of the experimental station serves as the core characterization method, providing rich data support for the overall experiment (Fig. 5) [6-8].

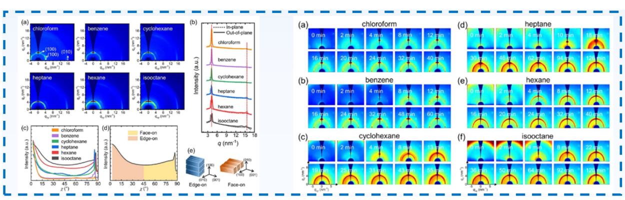

Figure 4: Solubility parameter regulation of D-A conjugated polymer condensed state structure.

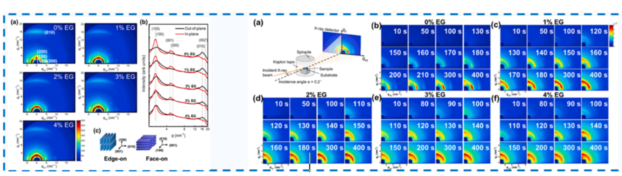

Figure 5: Additives regulate the crystallization behavior of D-A conjugated polymers.

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