New Apparatus & Upgrades

A Beamline Dedicated to Soft Matter Research has been Now Launched at BL03XU

The "Frontier Soft Matter Beamline (FSBL)" constructed by the Advanced Softmaterial Beamline Consortium consisting of 19 research groups from both commercial corporations and universities was launched at **BL03XU** on February 4, 2010. FSBL will open the door to new activities aiming to further advance polymer science and develop soft matter/new polymeric materials by fully utilizing the sophisticated light source capabilities of SPring-8. It is the first full-fledged strategic concept in the world that promotes the collaborative use of a radiation facility among a government, businesses, and universities

FSBL is equipped with X-ray diffraction/scattering measurement systems that can be used for high-speed simultaneous measurements of the hierarchical structures of bulk and thin-film samples of soft matter at a scale of several nanometers to several hundred nanometers. This beamline employs the standard SPring-8 in-vacuum undulator (magnet material: NdFeB, periodic length: 32 mm, number of periods: 140, total length: 4.5 m, available energy: 5 keV ~ 18 keV) as the light source and the cryogenic-cooling double-crystal monochromator. Rh- and Pt-coated mirrors (horizontal and vertical) are used to focus and eliminate the higher harmonics of the undulator beam. The performance of the X-ray at the sample position is presented in Table 1.

The experimental hutches consist of the first and second hutches as presented in Fig. 1. The first hutch incorporates a measurement system that can be used to carry out grazing-incidence wide-angle X-ray diffraction (GIWAXD) measurements and also grazingincidence small-angle X-ray scattering (GISAXS) measurements when an X-ray is directly injected very close to the surface of a sample using a thin-film goniometer (horizontal placement type), time-resolved measurements with both GIWAXD and GISAXS, simultaneous measurements, and X-ray reflectometry. It is the only measurement system in Japan that can be used to unravel the dynamic structures of thin organic/polymer films and surfaces/interfaces in various external environments. It is expected to greatly contribute to improving the performance of polymeric materials/soft matter for use in a wide variety of fields (e.g., electronic devices, batteries/cells, adhesives/coating, printing, and biomaterials) and products such as organic ELs, organic FETs, organic memory, organic fuel cells, and organic solar cell materials. The second hutch incorporates a system that can be used to carry out small-angle X-ray scattering (SAXS) measurements, wide-angle X-ray diffraction (WAXD) measurements, and simultaneous time-resolved measurements that combine SAXS/WAXD and various other different measurements (Fig. 2). If necessary, a kinematic mount can installed in the first hutch at the space of 3 m length × 3 m width × 4 m height, which enables to observe in situ structural changes in real processing such as fiber spinning and film drawing. We are planning to install even more advanced optical and measurement systems to observe time-fluctuation in structure and nano- or meso-structures.

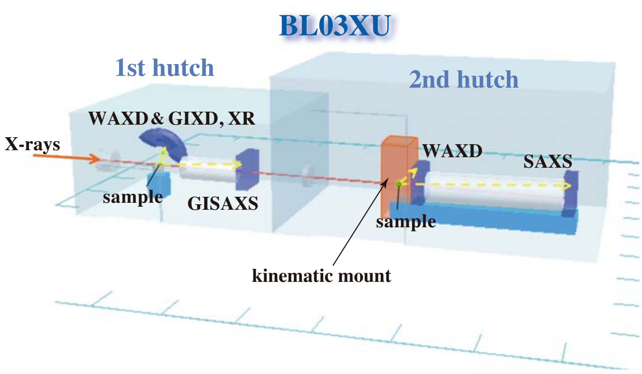

Fig. 1. Outline of the experimental hutches.

The first test measurement was carried out using one of the ultrafine prototype vinylon fibers produced in the early stage (around 1955) of its commercialization. Vinylon was the first synthetic polymer fiber entirely produced in Japan. The newly constructed beamline can be used to instantly produce pictures of fibers and "nano-filaments." Figure 3(b) shows an X-ray scattering image visualized on a two-dimensional digital detector after irradiating it with X-rays for no more than 20 s. As can be seen in the figure, the image is very sharply defined. The image clearly reveals very weak scattering at an extremely small scattering angle, and thus also revealing that the fiber has an orderly structure with a microcrystalline region formed by polymer molecules and a noncrystalline (amorphous) region (similar to irregular glass structure) alternating at 18.5 nanometer intervals. The sharp clear X-ray scattering pattern provided in Figure 3(c) (fiber diffraction pattern) reveals that it has a molecular orientation where the polymer chains in the microcrystal

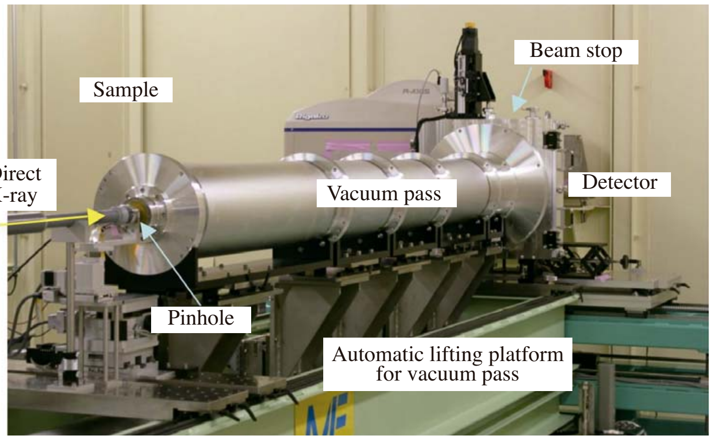

Fig. 2. SAXS measurement in the second experimental hutch (length of vacuum path: 3 m).

have been arranged in the axial direction of the fiber at amicroscopic level of accuracy. Japanese vinylon was manufactured half a century ago and demonstrated, at the molecular level, the high level of technology used to produce polymer fibers in those days.

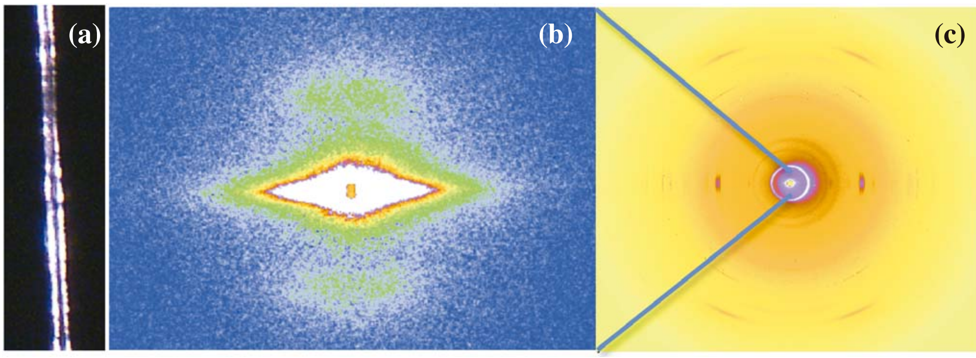

Fig 3. SAXS **(b)** and WAXD **(c)** patterns of a single vinylon fiber **(a)** approx. 15 μm in diameter. Measurement conditions -- wavelength: 0.1 nm; vacuum path length: (b) 3 m and (c) 30 cm; detector: (b) Image Intensifier +CCD detector (ORCA R2) (Hamamatsu Photonics K.K.) and (c) Imaging Plate system (R-AXIS VII) (Rigaku Corporation); sample: vinylon prototype manufactured in late 1950s to early 1960s, contributed (loaned) to the Institute for Chemical Research of Kyoto University (Prof. Toshiji Kanaya) by a descendant (member of the Takatsuki-kai group) of Professor Ichiro Sakurada, Kyoto University who succeeded in synthesizing vinylon for the first time in Japan. (b) shows the scattering pattern that was revealed by measuring it at a favorable angle and resolution around the center of (c).

Table 1. X-ray performance of BL03XU

Energy range	6 to 35 keV (2.0 to 0.36 Å)
Energy resolution	$\Delta E/E < 10^{-4}$
Photon flux	$> 10^{13}$ (without slits)
Beam size	170 μm (H) × 80 μm (V)

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